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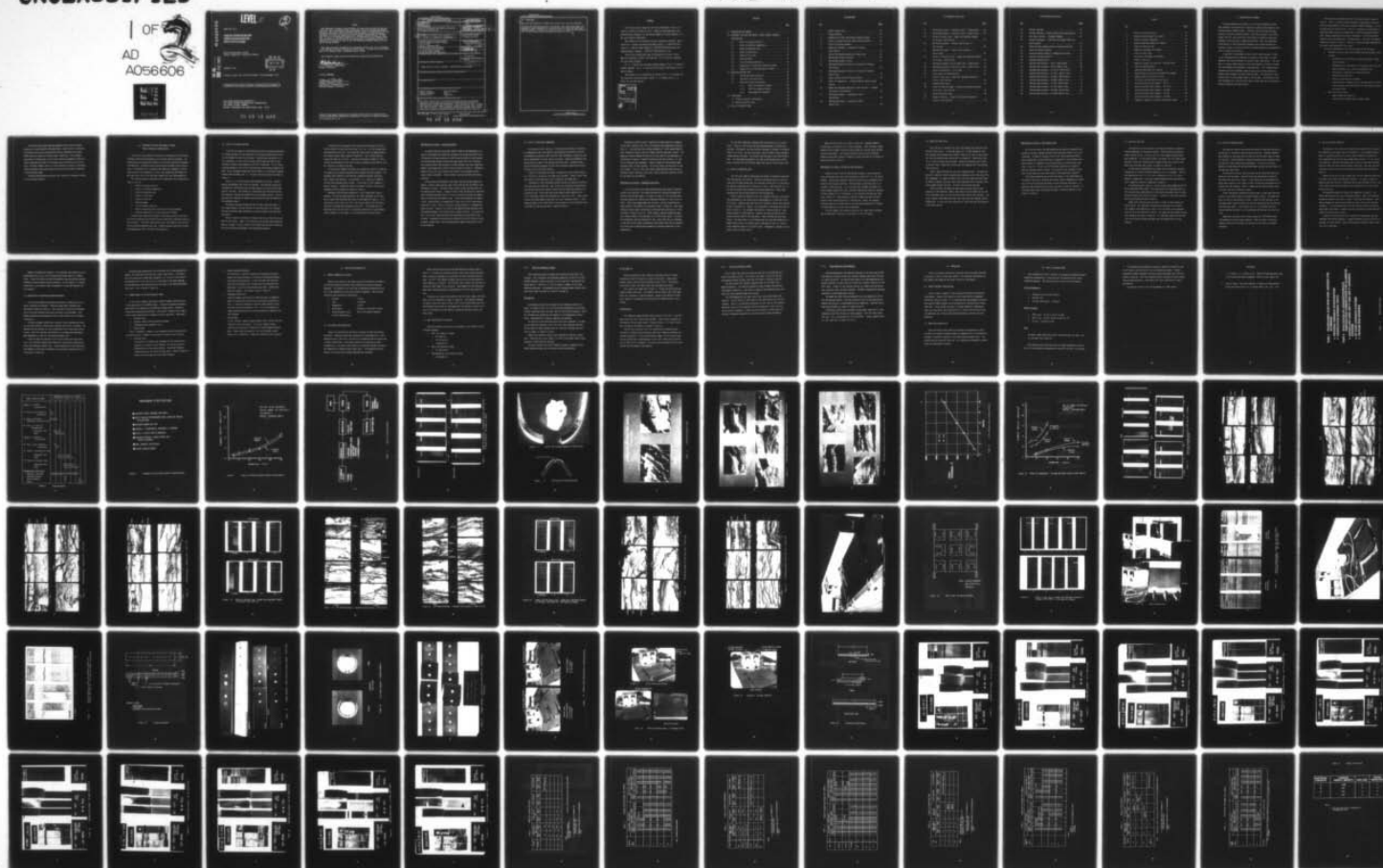
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JAN 78 M C LOCKE, R E HORTON, J E MCCARTY F33615-73-C-5171

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ANODIZE OPTIMIZATION AND
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REPAIR APPLICATIONS

Seattle, Washington 98124
Boeing Commercial Airplane Company



JANUARY 1978

Interim report for period December 1976-September 1977

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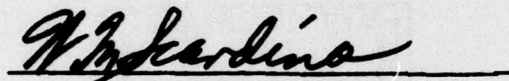
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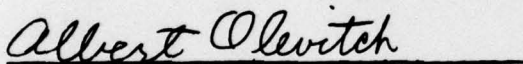
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W. M. SCARDINO (AFML/MXE)
Project Engineer

FOR THE COMMANDER



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report covers Task I of a two-task follow-on program to investigate phosphoric acid non-tank anodize process optimization and evaluation of adhesive/surface preparation combinations. Work completed in Task I included investigating non-tank anodizing process variables of voltage, time, temperature, and rinse delay. Other parameters studied included the effect of anodizing over titanium and aluminum fasteners, battery anodizing, anodizing		

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mode, and identification of common errors occurring in non-tank anodizing.

Following the anodizing variables investigation, bond verification tests were conducted to assess bondability of representative RT, 250°F, and 350°F cure adhesive systems. This work will serve as a basis for selecting the phosphoric acid non-tank anodize process parameter/conditions for Task II.

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FOREWORD

This interim report summarizes the work accomplished in Task I, an "add-on" to Contract F33615-73-C-5171, "Adhesive Bonded Aerospace Standardized Repair Handbook," by the Boeing Commercial Airplane Company, P. O. Box 3707, Seattle, Washington.

Other phases of this contract have been reported as follows: Phase I report was a limited distribution document; Phase II -- AFML-TR-75-158; Phase III -- AFML-TR-76-65; Phase IV -- AFML-TR-76-201/AFFDL-TR-76-131; Completed Repair Handbook -- AFML-TR-77-206/AFFDL-TR-77-139.

The work was accomplished under the sponsorship of the Air Force Materials Laboratory (Project 7381/Task 06). Mr. W. Scardino, AFML/MXE, was the project engineer.

Mr. J. E. McCarty was the Boeing program manager, with R. E. Horton as project engineer. Mr. M. C. Locke was the principal investigator for the add-on program.

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1.0 INTRODUCTION AND SUMMARY

It became apparent during Phase II of the Repair Handbook contract that additional investigation was required to optimize the phosphoric acid non-tank anodize (PANTA) process. Excellent results had been obtained using this surface preparation method. The size and scope of the effort, however, did not allow sufficient investigation to establish processing parameter limits relative to anodizing potential, time, and temperature. Investigation of these processing parameters and related variables was required in order to bring this surface treatment method to the operational level for repair applications.

In addition to development of the non-tank anodize process in Phase II, an investigation was conducted to evaluate a large number of repair adhesives and cleaning methods for possible repair application. This work was of a cursory nature because of the large number of variables involved. This work needed to be expanded in selected areas. This included further evaluation of prime candidate adhesive systems and surface preparation methods, bond strength, and bond durability data. This additional investigative work is being accomplished in this program. The additional data and information will facilitate the repair process and form a data base for material selection and repair design.

The current add-on program consists of two major tasks as shown in figure 1. Task I is aimed at process parameter investigation and optimization on the phosphoric acid non-tank anodize (PANTA) procedure. It also includes bond verification tests. Task II is focused on an evaluation of adhesive/surface preparation combinations, including vacuum bag bonding under reduced temperature and pressure conditions. The two-task add-on program is being performed over a 19-month period. The tasks and program schedule are shown in figure 2. The Task I program has been completed and the results are presented in this report.

The work accomplished in this task consisted of the following:

- o Phosphoric acid non-tank anodize (PANTA) process variables investigation
 - Determination of the effects of anodize potential, temperature, and time
 - Determination of the effects of rinse delay, part size, and anodizing modes (e.g., vertical, horizontal, etc.)
 - Evaluation of anodizing clad versus bare alloy
 - Anodizing with batteries
 - Effect of anodizing over fasteners
 - Identification of common errors in the application of PANTA
 - Oxide thickness characterization with the scanning electron microscope (SEM)
- o Bond verification tests
 - Bond strength and durability
 - Verification of PANTA process variable limits

Test results have shown that the phosphoric acid non-tank anodize process can be satisfactorily effected under a wide variety of conditions. Satisfactory results were obtained when process parameters were varied within practical ranges to simulate repair conditions. This included variations in voltage from 2 to 10 volts; anodizing temperature from 40 to 100°F; anodizing time from 5 to 20 minutes; and rinse delay up to 10 minutes. We noted, however, that it is more difficult to obtain a consistent, uniform anodized surface when anodizing in the vertical mode or underside in the horizontal mode.

The work accomplished during this Task I period is discussed in detail in the following sections.

2.0 PHOSPHORIC ACID NON-TANK ANODIZE (PANTA)

PROCESS VARIABLES INVESTIGATION

This part of the program was aimed at an investigation of the process variables affecting the phosphoric acid non-tank anodize procedure. The objective was to evaluate the effects of the various processing parameters on bondability. Bondability was assessed using the wedge crack extension test. In addition, prior to bonding into wedge test assemblies, the anodized surfaces were inspected for color, tape tested with 3M Company No. 250 tape, and a strip (1/8-inch wide) removed for later SEM examination.

The following process parameters were selected for this investigation (Task I, Part A):

- o Effect of voltage variation
- o Effect of anodizing temperature
- o Effect of anodizing time
- o Effect of rinse delay
- o Effect of part size
- o Anodizing modes
- o Dry cell battery anodizing
- o Effect of anodizing over titanium and aluminum fasteners
- o Identify common errors in the application of PANTA

Following the processing parameters investigation, bond verification tests were conducted using selected surface preparation conditions based on Part A results. In bonding verification tests, both 2024-T3 clad and 7075-T6 bare aluminum adherends were used. Adhesive systems evaluated included room-temperature, 250°F, and 350°F cure materials.

2.1 EFFECT OF VOLTAGE VARIATION

This task was aimed at investigating the effect of anodizing potential or voltage variation on the phosphoric acid non-tank anodize process relative to bondability and oxide thickness. Anodizing was carried out at 1, 2, 4 (baseline), 6, and 10 volts on both 7075-T6 bare and 2024-T3 clad aluminum at ambient room-temperature (70-75°F) conditions. For all tests, a gelled phosphoric acid (10-12% concentration) from Products Research was used. Basic procedures established during Phase II of the Repair Handbook contract were followed (ref. 1). The basic processing steps are shown in figure 3.

Two 6- x 6- x 1/8-inch panels were anodized at one time. Current density measurements were taken and recorded. The anodized surface was inspected for interference color and tape tested on one end using 3M Company No. 250 tape. The presence of interference color and positive tape test generally indicate the bondability of the treated surface. Positive tape test was indicated by adhesive pulling from the tape and adhering to the anodized surface.

The panels then were primed with BR 127 CIAP primer and cured. A strip approximately 1/8-inch wide was cut from each panel for scanning electron microscope (SEM) examination of oxide thickness and oxide characteristics.

Table 1 compares the effect of voltage variation on 7075-T6 bare and 2024-T3 clad alloys. Shown in this table are the current density measurements as voltage is varied, results of the tape test and color inspection, and oxide thickness measurements from SEM photomicrographs.

Current density measurements were taken during anodizing at the different selected voltages when two pieces of 6- x 6- x 1/8-inch wedge panels were anodized at one time. This setup was used so that one wedge assembly was surface treated under identical conditions. Thus, current density values are shown for 0.5 sq ft. The data are plotted in figure 4, illustrating the differences of the 7075-T6 bare as compared to the 2024-T3 clad as a function of voltage variation.

The panels were bonded into wedge test assemblies using FM 73 adhesive. Five wedge crack extension specimens were prepared from each assembly. The specimens were exposed to 120°F/95-100% condensing humidity conditions. Crack lengths were measured. Failure modes, i.e., cohesive or adhesive failure, were evaluated after 30 days. A flow chart of this sequence is shown in figure 5. Wedge test results are shown in table 2, and the corresponding failure surfaces are shown in figure 6.

Specimens (7075-T6 bare) anodized at 1 volt failed after 4 hours exposure. No failures were observed on all other specimens in this task and no significant difference was found in the wedge test results. It is interesting to note that the 1 volt, 7075-T6 bare panels also failed the tape tests, although they showed positive color. This agrees with our experience that positive color is a necessary indicator on a good bonding surface; however, it may appear on an unsatisfactory surface as well.

SEM Analysis of Oxide -- Voltage Variation

A scanning electron microprobe (Model 131000-32 SEM Quantometer) was used to investigate the oxide layer thickness and oxide characteristics as a function of voltage variation on 7075-T6 bare and 2024-T3 clad aluminum. The 1/8-inch-wide aluminum strips were bent around 180° until fractured. The fractured halves were mounted on an SEM specimen stub using conductive cement, then coated with a layer of gold as shown in figure 7. Photomicrographs of the oxide cross-section were taken at varying magnification levels (500X to 20,000X). Figure 8 shows a typical specimen and viewing direction.

Oxide thickness measurements were taken from the 20,000X photomicrographs. Figure 9 shows typical views of the 7075-T6 bare and 2024-T3 clad anodized at 10 volts. The CIAP primer coating, oxide, and base metal can be observed rather easily. Figures 10 and 11 show the effects of anodizing under different voltages (from 1 to 10) on the oxide thickness and appearance on 7075-T6 bare and 2024-T3 clad. Little oxide formation is noted at 1 volt. Oxide buildup is increased at 2 volts, but is still quite nonuniform until 4 volts. From 4 to 10 volts, oxide thickness is increased and rather uniform. Figure 12 is a plot of the measured oxide thickness from the photomicrographs, and plotted versus voltage variation. The values for the 1- and 2-volt conditions were from measurements on the 2024-T3 clad surfaces. Inadequate resolutions were obtained on the 7075-T6 bare surface at these voltage levels, and oxide appearance suggests nonuniform or thin oxide formation.

2.2 EFFECT OF ANODIZING TEMPERATURE

The purpose of this task was to investigate the effects of anodizing at temperatures other than ambient room-temperature conditions (70-75°F). Two temperature extremes (100°F and 40°F) were selected as conditions that may be encountered in actual repair situations. Anodizing procedures used were similar to those in the voltage variation study. Substrates were heated with heating blankets for the 100°F anodizing condition and cooled with dry ice for the 40°F condition.

Figure 13 illustrates the effect of anodizing on the current density as a function of voltage at the temperature extremes. Tables 3 and 4 show the results of the tape test, color inspection, and wedge test.

Processing at 100°F presented problems related to applying the gelled acid and setup for anodizing. Upon coating the aluminum with gelled acid, the etching reaction was rapidly taking place and there was drying out of the gelled acid. Due to the high current density when anodizing at 4 volts, additional panels were anodized at 2 volts and 1 volt. These latter surfaces provided negative tape test and color inspection results. These wedge specimens also failed within 4 hours exposure to 120°F/100% RH, which correlates to the tape and color inspection results.

Anodizing at 40-45°F shows a lowering of current density as compared to the 100°F condition (fig. 13) or the ambient room-temperature condition (fig. 4). We noted that the surfaces anodized at 2 volts showed negative color inspection results and only slightly positive tape test results. The wedge test results showed failures only on the 2024-T3 clad specimens, as illustrated in table 4. Sporadic failures can be seen in the 2-, 4-, and 10-volt anodized clad specimens. Figure 14 shows the disassembled wedge specimens after 30 days exposure. The failure modes correlate well to the crack growth. That is, long crack length showed adhesive-type failure at the metal-primer interface, while short crack growth gave cohesive failure at the center of the bondline.

SEM Analysis of Oxide -- Temperature Variation

As in the previous task, SEM photomicrographs were taken of anodized surface cross-sections. An attempt was made to correlate the wedge test results with the SEM surface. As shown in figure 15, little oxide formation was observed for 7075-T6 bare adherends anodized at 1 and 2 volts at 100°F. These also showed wedge test failures. Similar characteristics were evident on the 2024-T3 clad specimens shown in figure 16. More oxide formation can be seen on both bare and clad specimens anodized at 4 volts, as shown in figures 15 and 16. These surfaces produced acceptable wedge test results except in one specimen in the 7075-T6 bare group. No plausible explanation could be advanced for this, other than that all specimens in the 7075-T6 bare group had moderate crack growth after 30 days exposure. This points out a problem when attempting to anodize substrates at high temperatures.

For the 40°F conditions, anodizing was carried out at 2, 4, and 10 volts. Figures 17 and 18 show the SEM photomicrographs of oxide cross-sections. Here again, the oxide buildup increases as voltage is increased from 2 to 10 volts. We note from table 4 that the 2024-T3 clad specimens showed sporadic failures. No failures were observed on the 7075-T6 specimens. The SEM photomicrographs do not show oxide characteristic differences to account for the different wedge test results on bare versus clad.

2.3 EFFECT OF ANODIZING TIME

This task was aimed at determining the effect of anodizing time other than the baseline (10 minutes). Anodizing procedures similar to earlier tasks were followed except that the times were varied from 1 to 20 minutes. The applied voltage was held constant at 4 volts. Time intervals of 1, 5, 15, and 20 minutes were selected to evaluate bondability. These times represent the low and high variations from the baseline.

Table 5 shows the tape test and color inspection results, oxide thickness measurements, and current density measurements as a function of anodizing time. Negative tape test and color inspection results were obtained on parts anodized for 1 minute. This correlates to the wedge test results shown in table 6. All specimens anodized for 1 minute failed in 1 hour during exposure to 120°F/100% RH. Sporadic failures occurred on the 5-minute anodized, 2024-T3 clad specimens. These failures were not unexpected. The short time (1 to 5 minutes) does not allow the formation of a stable porous oxide. As noted by other investigators (ref. 2), initial oxide formation results in a barrier layer. Subsequently, formation of the porous oxide film takes place.

Wedge failure surfaces are shown in figure 19. Complete adhesive failure mode is obvious on the 1-minute specimens. Other specimens showed cohesive failure modes, indicating good bondability, except those sporadic failures noted above. The longer anodizing times (15 and 20 minutes) showed no measurable change as compared to those anodized for 10 minutes or baseline condition (table 2).

SEM Analysis of Oxide -- Anodizing Time Variations

Figures 20 and 21 show the SEM photomicrographs of oxide cross-sections on the bare and clad alloys anodized at 4 volts for 1, 5, 15, and 20 minutes. These times represent low and high durations as compared to the baseline (10 minutes) condition. Oxide buildup as anodizing time is increased is quite apparent. It can be seen that specimens anodized for 1 minute showed comparatively thin, sparsely formed oxide layers as compared to those formed at 15 or 20 minutes, or the baseline 10-minute case (see figs. 10 and 11). These surfaces resulted in wedge test bond failures within 4 hours during exposure to 120°F/100% RH. Except for sporadic failures occurring in three 2024-T3 clad specimens anodized for 5 minutes, no other wedge test failures were encountered.

The data suggest that anodizing should be for longer than 5 minutes, and no observable difference can be shown for 10 to 20 minutes.

2.4 EFFECT OF RINSE DELAY

This task was to establish the time limit between the anodizing cycle and the rinsing cycle. It has been shown (ref. 2) that phosphoric acid will dissolve the aluminum oxide formed if the acid is left on the surface for long time periods after the current is turned off. Rinse delay times of 2, 5, and 10 minutes were selected. Anodizing procedures were similar to earlier tasks, except that rinsing does not commence until the selected time lag period.

Table 7 shows the tape test and color inspection data. The tape test and color inspection results progressively degraded as the rinse delay time was lengthened. In comparing these results with the wedge test data shown in table 8, however, only the 7075-T6 bare specimens with 10 minutes rinse delay showed sporadic failures. No other failures were observed.

Figure 22 shows the wedge specimen bonded surfaces after 30 days exposure to 120°F/100% RH. Nearly 100% cohesive failure modes can be seen on all specimens except for the 10-minute delay 7075-T6 bare specimen. The latter showed a 100% adhesive failure mode resulting from improper surface preparation. In this case, the long delay of rinse time had contributed to a poor bond surface.

SEM Analysis of Oxide -- Rinse Delay Times

As in earlier tasks, the SEM examination was made on fractured cross-sections of primed, anodized surfaces with rinse delay times of 2, 5, and 10 minutes. As shown in figures 23 and 24 for 7075-T6 bare and 2024-T3 clad, respectively, oxide dissolution is apparent for the 10-minute rinse delay surfaces as compared to the 2-minute delay or no delay cases (see figs. 10 and 11). In fact, for 7075-T6 bare, practically no oxide to sparsely scattered oxide is noted. This condition also resulted in adhesive failure modes and long crack growth in the wedge tests (table 8).

The oxide dissolution due to the rinse delay is less significant on the 2024-T3 clad alloy, although here too we noted an appearance of loosening of the oxide from the base metal for the 10-minute rinse delay. In addition, when comparing the no delay case (figs. 10 and 11) and the 2- to 10-minute delay cases, the gradual decrease in the oxide density or uniformity is obvious.

2.5 EFFECT OF PART SIZE

The objective of this task was to evaluate the effects of anodizing aluminum adherends for large-area repairs. Earlier work had focused on panels ranging from 6- x 6-inch to 14- by 17-inch wedge and metal-metal peel assemblies. In the current effort, two panels, 24 x 24 inches, were anodized. Electrical contacts were arranged on the four corners of the panels and stainless steel screen, as shown in figure 25.

Current density measurements were taken and the electrical contacts removed one at a time at selected intervals (i.e., 2-3 minutes). The current density was 1.45, 1.4, 1.3, and 1.2 amps/sq ft for 4, 3, 2, and 1 contacts, respectively. All areas checked showed positive tape test and color inspection results as summarized in table 9.

The anodized panels (two 24- x 24-inch pieces) were bonded with FM 73/BR 127 adhesive/primer system. Nine wedge panels (45 specimens) were cut from selected areas representative of the anodizing contact areas or areas various distances away from the contacts. Figure 26 illustrates the specimen cutting diagram and panel location.

Wedge crack extension data are shown in table 10 after exposure to 120°F/100% RH. No significant difference was observed in the results. This suggests that adherends as large as 24 x 24 inches can be anodized using as few as one electrical contact. The wedge specimen bonded surfaces after exposure are shown in figure 27. All specimens shown had 100% cohesive failure when the wedge specimens were disassembled after 30 days exposure.

2.6 EFFECT OF ANODIZING MODES

This task was aimed at evaluating the effects of anodizing surfaces in the vertical or horizontal (both surfaces) positions. Aluminum adherends were anodized in simulated vertical position and horizontal modes as shown in figure 28. Various methods of securing the gelled acid saturated gauze and stainless steel screen were investigated. For vertical surfaces, methods of taping down the top and bottom edge, top edge and two sides, and top edge only were tried.

On horizontal surfaces, anodizing both top and bottom and bottom surfaces only was evaluated. Some difficulty was experienced in both the horizontal and the vertical cases. It was found that it was necessary to apply some pressure to the screen in order to keep the gauze/paste acid in contact with the aluminum. Table 11 summarizes the anodizing mode study, tape test, and color inspection results.

Visual observation indicated nonuniform areas in some cases when anodizing in the vertical mode or horizontal bottom surface. The wedge test data in table 12 show sporadic failures. These failures occurred in the horizontal mode, bottom surfaces, and vertical mode where the top edge and two sides were taped. In the latter case, it is postulated that this taping technique resulted in the screen/gauze pulling away from the aluminum.

Wedge test specimens after 30 days exposure to 120°F/100% RH were disassembled and failure modes examined. These are shown in figure 29. Complete adhesive failure modes are obvious on the large crack growth specimens.

2.7 DRY CELL BATTERY ANODIZING

The purpose of this task was to determine the feasibility of using dry cell batteries as the power source for anodizing. This procedure would be useful in preparing surfaces for repairs under remote field conditions. The investigation was carried out on wedge panels using 2-, 4-, 6-, and 12-volt batteries. Anodizing procedures used in earlier tasks were followed. Figure 30 shows the test setup for battery anodizing. Tape test and color inspection results and the current density and voltage data are shown in table 13.

Except for the 2-volt case in which two 1.35-volt batteries were used, all others were single, off-the-shelf batteries. As shown in table 13, negative tape test and color inspection results were obtained in the 2- and 4-volt cases, while positive results were obtained on the 6- and 12-volt anodized surfaces.

Corresponding wedge test results are summarized in table 13. No panels were bonded on the 2-volt anodized surfaces. Both 4-volt anodized assemblies showed failure within 1 hour of 120°F/100% RH exposure. Satisfactory wedge test results were obtained on the 6- and 12-volt anodized specimens. It is postulated that the 4-volt battery did not provide adequate power (only about 0.5 amp/5 sq ft) for the anodizing time duration to develop the proper oxide surface.

Figure 31 illustrates the failure surfaces on the battery anodized specimens after 30-day exposures. Complete adhesive failure on the 4-volt specimen is rather obvious. Nearly 100% cohesive failure is seen on the 6- and 12-volt specimens.

Based on the wedge test results, it is concluded that anodizing can be accomplished using a 6- or 12-volt battery when making repairs at remote sites. It must be pointed out that the batteries must provide the required sustaining current capacity during anodizing. In this regard, it is postulated that a 4-volt battery might be adequate if it had the required current capacity.

2.8 ANODIZING ON TITANIUM AND ALUMINUM FASTENERS

This task was aimed at evaluating the effects of anodizing over fasteners (titanium and aluminum). Adhesive bonded metal laminates were fabricated as shown in figure 32. Four titanium 1/4-inch Hi-Lok fasteners and six 1/4-inch aluminum rivets were installed in the specimens. The bonded specimens were topcoated with BR 127 CIAP primer prior to fastener installation.

Following fastener installation, one-half of each specimen was abraded, non-tank anodized, rinsed, dried, reprimed with BR 127, and baked. The anodized area was checked for acid contamination with litmus paper during rinsing. With this specimen configuration, no problems were encountered when attempting to rinse off the gelled phosphoric acid.

Figure 33 shows the specimens after 14 and 30 days salt spray exposure. No significant changes were observed on the non-tank anodized area versus the untreated, control area. Closer examination of the countersunk area showed no significant difference in the corrosion characteristics, as illustrated in figure 34.

Following these examinations, the 0.020-inch skin of each specimen was peeled. No significant difference was found in the results. The control half and treated half showed peel strength of 111 lb-in./2.1-inch width on one specimen and 111 and 105 lb-in./2.1-inch width on the other specimen. Failure modes were 100% cohesive with no corrosion in the bondline/fastener hole areas. This is shown in figure 35.

2.9 COMMON ERRORS IN THE APPLICATION OF PANTA

In this task, attempts were made to identify common recurring errors in the application of the PANTA process. Incorrect processing procedures were tried in the laboratory and observable effects noted. Corrective actions were proposed and evaluated. Four typical situations were identified as high probability for frequency of errors occurring. These were:

- o Reversed leads
- o Stainless steel screen contacting aluminum
- o Inadequate gelled phosphoric acid
- o High voltage

Table 14 presents a summary of the observable results and applicable corrective actions. Comments on the tests and results are as follows:

- o Reversed Leads

The positive (+) terminal was connected to the screen and the negative (-) terminal to the aluminum. The obvious reaction observable was on the screen surface. The surface showed no interference color and failed the tape test. Figure 36 shows the effect of this mistake and the correct procedure.

o Screen Contacting Aluminum

This situation is typically caused by not extending the gauze beyond the screen periphery, or insufficient separation between the screen and the aluminum. In the case where the stainless steel screen (cathode) was in contact with the aluminum while anodizing, a surge in current density was noted. Burned spots were also evident. This effect is seen in figure 37.

o Inadequate Gelled Acid

Figure 38 compares the results of anodizing when an inadequate amount of gelled acid was applied to the properly anodized surface. It should be pointed out that the gauze should be saturated with the gelled phosphoric acid. It is also important to reapply additional gelled acid during anodizing to replenish the acid consumed.

o High Voltage

In this instance, higher voltage (greater than 10 volts) than the baseline 6 volts was applied. The results showed vigorous reaction with high (over 10 amps/sq ft) current density measurement. Proper anodizing potential (4-6 volts) showed current density of 1-7 amps/sq ft. Figure 39 illustrates this situation.

3.0 VERIFICATION BONDING TEST

3.1 PROCESS PARAMETERS SELECTION

Based on the results of the wedge tests and SEM analysis, phosphoric acid non-tank anodize conditions were selected for preparing aluminum adherends. The purpose of this task was to demonstrate process verification of the phosphoric acid non-tank anodize procedures. The following conditions were selected for application:

- o Voltage: 6 volts
- o Time: 10 minutes
- o Temperature: 70-75°F
- o Rinse delay: No delay or less than 1 minute
- o Gelled phosphoric acid PR-50 from Products Research
(10-12% concentration)

3.2 TEST MATRIX AND FABRICATION

Table 15 illustrates the test matrix selected for bond verification evaluation. Adhesive systems were selected to be representative of room-temperature cure, 250°F cure, and 350°F cure categories that are being used in repair applications. Tests were selected to provide data on bond strength (e.g., lap shear), bond durability (sustained stressed lap shear), and compatibility (wedge and metal-metal peel). The comparative performance on clad versus bare aluminum adherends was evaluated.

Panels were fabricated using the adhesive/primer systems shown in table 15. Following the anodizing process, panels were primed and baked. Panel curing was completed in an autoclave for 250°F and 350°F cure systems. For 250°F cure adhesive systems, the cure cycle was 90 minutes at 250°F under 50 psi pressure. For the 350°F cure system, cure cycle was 60 minutes at 350°F under 50 psi pressure. Room-temperature cure bonds were made under vacuum bag pressure (approximately 10-12 psi) and cured for 7 days.

Following cure, panels were machined into lap shear, wedge, and metal-metal peel test specimens as shown in figure 40. Environmental exposure included 14 days salt spray (5% NaCl, 95°F) for the metal-metal peel test, 120°F/100% RH for 30 days for the wedge test, and 60 days or time to failure at 120°F/100% RH for the 1200-psi and 600-psi sustained stress lap shear tests.

3.3 BOND VERIFICATION TEST RESULTS

Bond verification test results are discussed in this section in the following sequence:

- o 250°F cure adhesive systems
 - FM 73/BR 127
 - AF 127-3/BR 127
 - EA 9601/BR 127
- o 350°F cure adhesive systems
 - AF 130/EC 2333
- o Room-temperature cure adhesive system
 - EA 9320/BR 127

3.3.1 250°F Cure Adhesive Systems

Three candidate adhesive systems were selected for the 250°F cure category. FM 73 represents new-technology adhesives, while AF 127-3 and EA 9601 represent systems currently in use at the ALC's (San Antonio and Warner Robins). Tables 16, 17, and 18 present a summary of the three adhesives evaluated. Corresponding typical specimen bonded surfaces after the various tests are shown in figures 41 through 46.

FM 73/BR 127

Satisfactory results were obtained on this adhesive system in all tests. As shown in table 16, no failures were encountered in the 60-day, 1200-psi sustained stress lap shear test at 120°F/100% RH exposure. Residual strengths were determined and compared to room-temperature control tests. Approximately 5% strength reduction was obtained.

All results of the metal-metal peel tests were acceptable. No undercut corrosion was observed on the 14-day salt spray exposure specimens. Failure modes on these specimens were all cohesive, including the -67°F test, as shown in figures 41 and 42.

Wedge crack extension results were comparable to tankline treated data. There was very little (about 0.14 inch) crack growth after 30 days exposure in 120°F/100% RH conditions.

The data indicate that the FM 73/BR 127 system is compatible with PANTA treated surfaces, and satisfactory bonds were obtained.

AF 127-3/BR 127

Results obtained with this adhesive system were similar to those obtained with the FM 73 material, except in two areas. Longer crack growths (about 0.40 inch) were obtained on the AF 127-3. In addition, significantly lower -67°F metal-metal peel strengths were noted.

Test results are summarized in table 17. Figures 43 and 44 show the bonded surfaces after test. The adhesive failure mode on the -67°F metal-metal peel specimens is readily apparent. Based on the results of other tests, however, no compatibility problem with the PANTA surface is anticipated.

EA 9601/BR 127

This adhesive system provided results similar to AF 127-3. Low -67°F metal-metal peel test results were obtained. These values correspond to the adhesive failure modes shown in figures 45 and 46. All other results were considered satisfactory, as shown in table 18.

The peel test values at -67°F are significantly affected by many variables, including primer thickness, bake time, adhesive thickness, and surface preparation. In this test program, however, BR 127 primer thickness was controlled to approximately 0.2 mil with a bake cycle of 60 minutes at 250°F prior to bonding. The lower values obtained on AF 127-3 and EA 9601 are attributed to the adhesive.

3.3.2 350°F Cure Adhesive System

The AF 130/EC 2333 adhesive system was the 350°F cure candidate evaluated on the PANTA surface. Test results are shown in table 19, and the corresponding bonded surfaces after test are shown in figures 47 and 48.

The data reflect the inherent characteristics of this 350°F cure adhesive material: low fracture toughness or peel strength and excellent environmental durability. The latter is shown by the sustained stress lap shear and crack extension wedge test results.

The room-temperature lap shear control test values were lower than expected. Typical values of about 2500 psi have been obtained on FPL etch surfaces. There is no plausible explanation at this time for this anomaly. Additional tests will be conducted using this adhesive in Task II on different surface treatments. Comparison and correlation of these data will be made to determine compatibility with the various surface treatments.

3.3.3 Room-Temperature Cure Adhesive

The room-temperature cure adhesive evaluated in this part was EA 9320. Test results are shown in table 20, and bond surfaces after test are given in figures 49 and 50. The results were typical for room-temperature cure adhesive materials in that there was significant strength reduction for the 180°F tests. Except for one specimen failure, all others survived the 60 days sustained stress lap shear exposure. Residual strengths were obtained comparable to room-temperature controls.

The wedge test data indicate compatibility of the adhesive to BR 127 and the anodized surface. The crack extension after 30 days exposure was moderate and typical of room-temperature cure adhesives. Cohesive failure modes were observed, as shown in figures 49 and 50.

Satisfactory results were obtained on metal-metal peel tests at room-temperature and after 14-day salt spray exposure. The -67°F peel values were low. This is characteristic of this material. Typical supplier data for -67°F peel test is about 6 lb-in./in.

4.0 CONCLUSIONS

Specific conclusions relating to individual tasks have been presented in sections 2.0 and 3.0 with test results. The following conclusions are more general and summarize the overall results of the program.

4.1 PROCESS VARIABLE INVESTIGATION

Table 21 shows a summary of the variables and processing conditions investigated. Based on the results of this investigation, recommended processing limits are given. It is concluded that the phosphoric acid non-tank anodize (PANTA) process can be satisfactorily effected under varying conditions. These include variation in voltage potential, temperature, time, and rinse delay; over large parts (2- x 2-ft); vertical and horizontal underside; over titanium and aluminum fasteners; and using dry cell batteries.

4.2 BOND VERIFICATION TESTS

Bond verification tests made with selected room-temperature, 250°F, and 350°F cure adhesive systems showed no incompatibility in the mechanical strength or durability exposure on non-tank anodized bonded joints. The optimum anodize conditions used (sec. 3.0) produced environmentally durable bonds and reproducible surfaces.

5.0 TASK II FOLLOW-ON PLANS

Work scheduled for Task II consists of evaluation of adhesive/surface preparation combinations. A test plan will be prepared for Air Force review and approval. The test matrix will consist of the following:

Surface Preparation

- o Phosphoric acid non-tank anodize
- o PasaJell 105
- o FPL etch (optimized) -- baseline

Adhesive Systems

- o 250°F cure -- AF 127-3, FM 73, EA 9628
- o 350°F cure -- AF 130, FM 400, FM 300, AF 143
- o RT cure -- EA 9320, EA 934

Tests

Lap shear, wedge, metal-metal peel, sustained stress lap shear, and L/t lap shear (see figure 40)

Both autoclave and vacuum bag cures at reduced temperature and pressure will be evaluated and compared with the 250°F and 350°F cure systems.

The candidates were selected to provide a spectrum of materials typical of those in use at the ALC's and new technology systems. Surface preparation methods represent two hand cleaning techniques and a tank procedure as baseline. The tests selected provide a balance of bond strength data and bond durability. The results will form a data base for repair applications.

The results of Task II will be documented in a final report.

REFERENCES

1. J. E. McCarty, R. E. Horton, et al., "Adhesive Bonded Aerospace Structures Standardized Repair Handbook," AFML-TR-75-158, August 1975.
2. John W. Diggle, "The Anodic Behavior of Metals and Semiconductors -- Oxides and Oxide Films, Vol. 2," Marcel Dekker, Inc., N.Y., 1973.

TASK I PHOSPHORIC ACID NON-TANK ANODIZE FOR ALUMINUM

- PROCESS VARIABLE INVESTIGATION
- VERIFICATION BONDING TESTS

TASK II EVALUATION OF ADHESIVE/SURFACE PREPARATION COMBINATIONS FOR ALUMINUM

- AUTOCLAVE CURE 250° F AND 350° F ADHESIVE SYSTEMS
- RT CURE ADHESIVE SYSTEMS
- VACUUM BAG BONDING

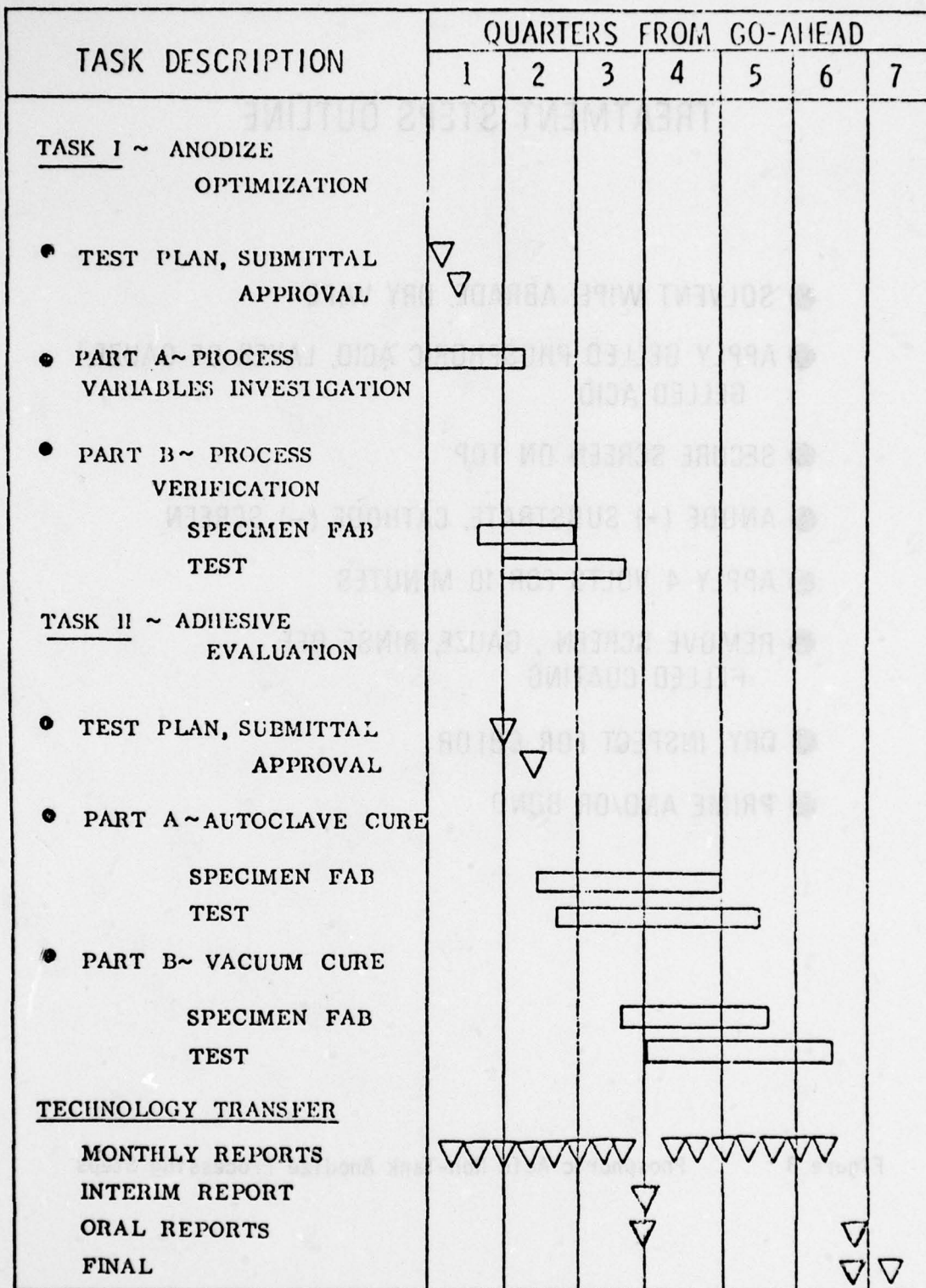


Figure 2 Program Schedule

TREATMENT STEPS OUTLINE

- SOLVENT WIPE, ABRASE, DRY WIPE
- APPLY GELLED PHOSPHORIC ACID, LAYER OF GAUZE, GELLED ACID
- SECURE SCREEN ON TOP
- ANODE (+) SUBSTRATE, CATHODE (-) SCREEN
- APPLY 4 VOLTS FOR 10 MINUTES
- REMOVE SCREEN , GAUZE, RINSE OFF GELLED COATING
- DRY, INSPECT FOR COLOR
- PRIME AND/OR BOND

Figure 3

Phosphoric Acid Non-Tank Anodize Processing Steps

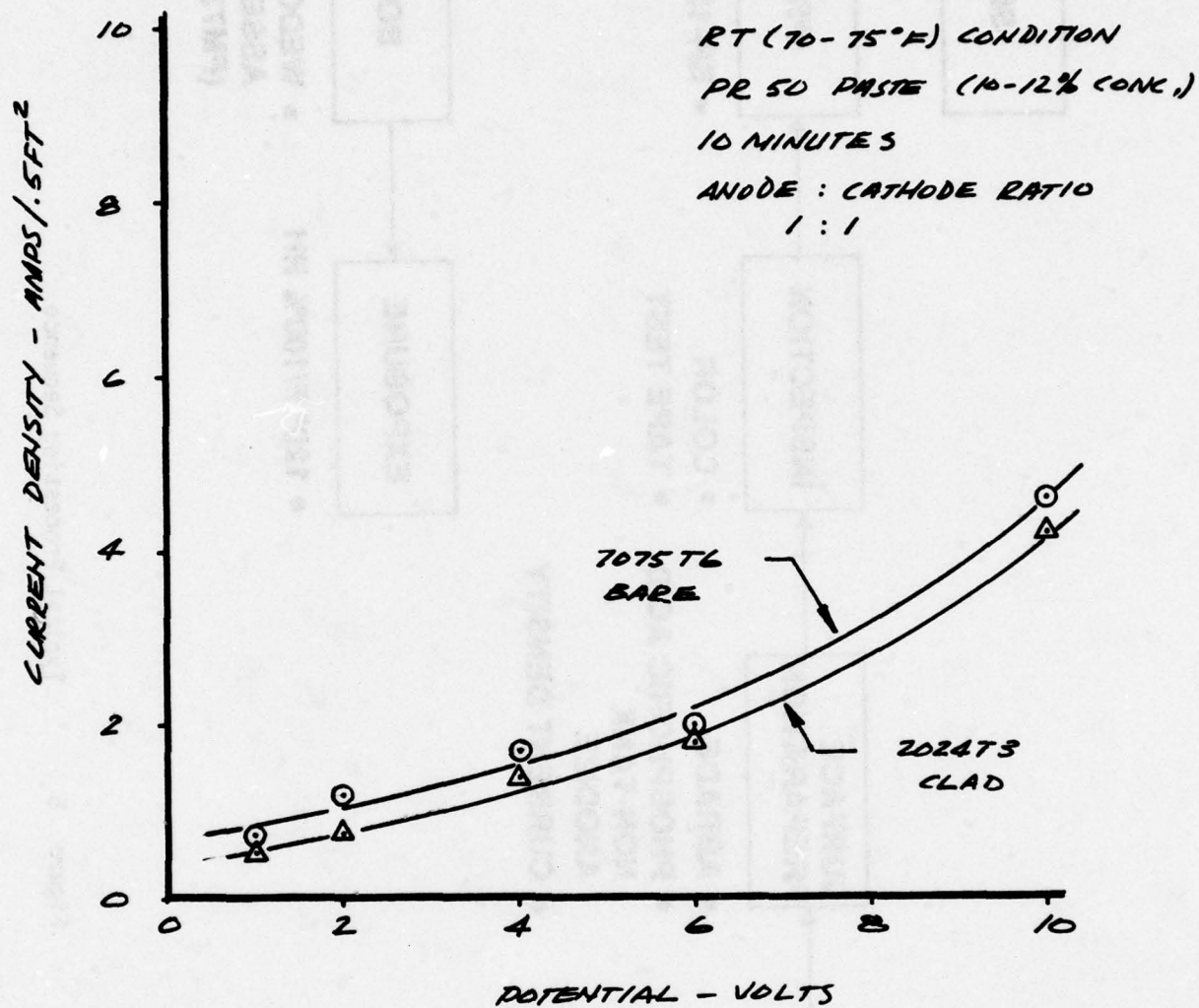


Figure 4 Effect of Voltage Variation versus Current Density

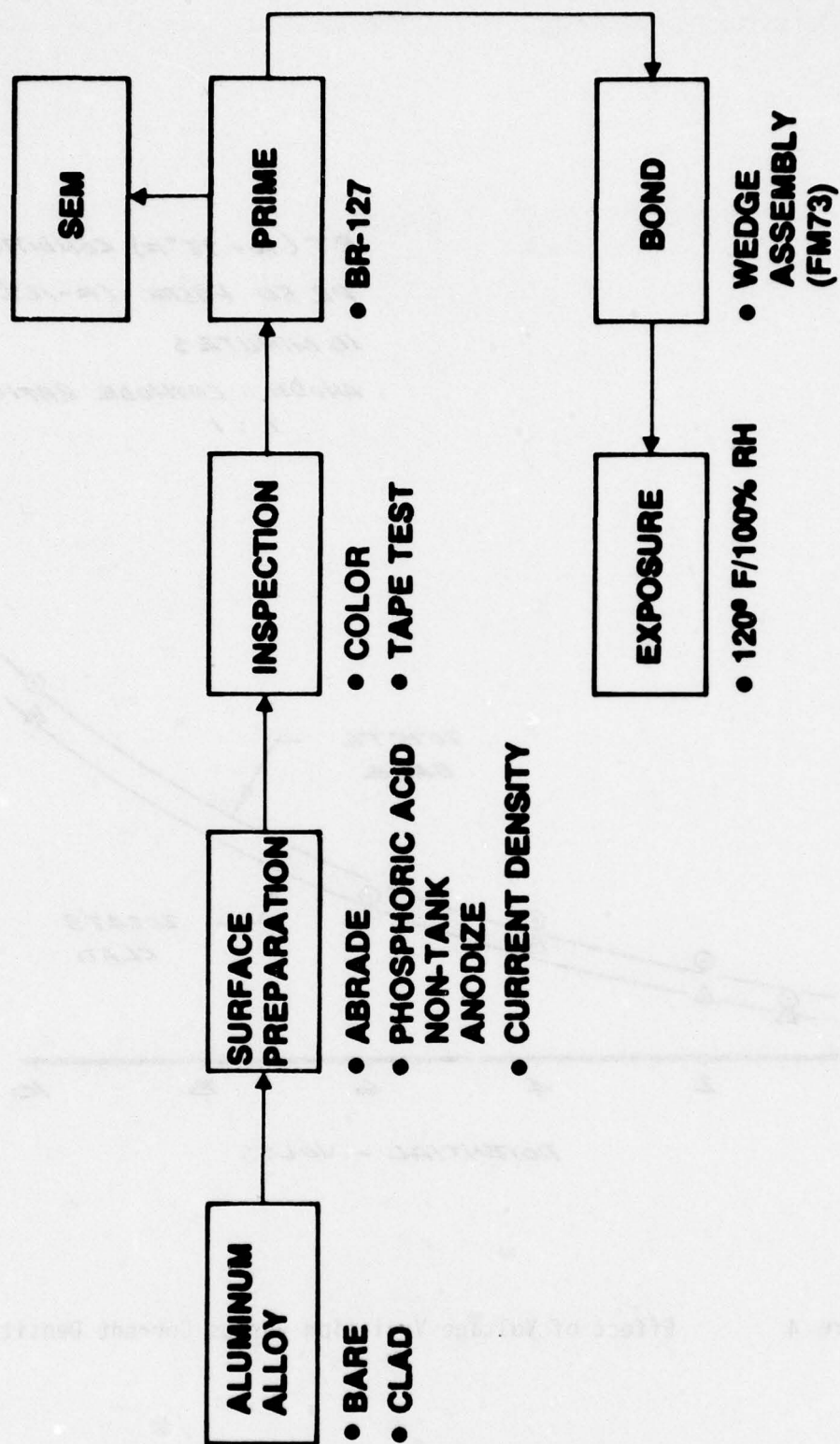
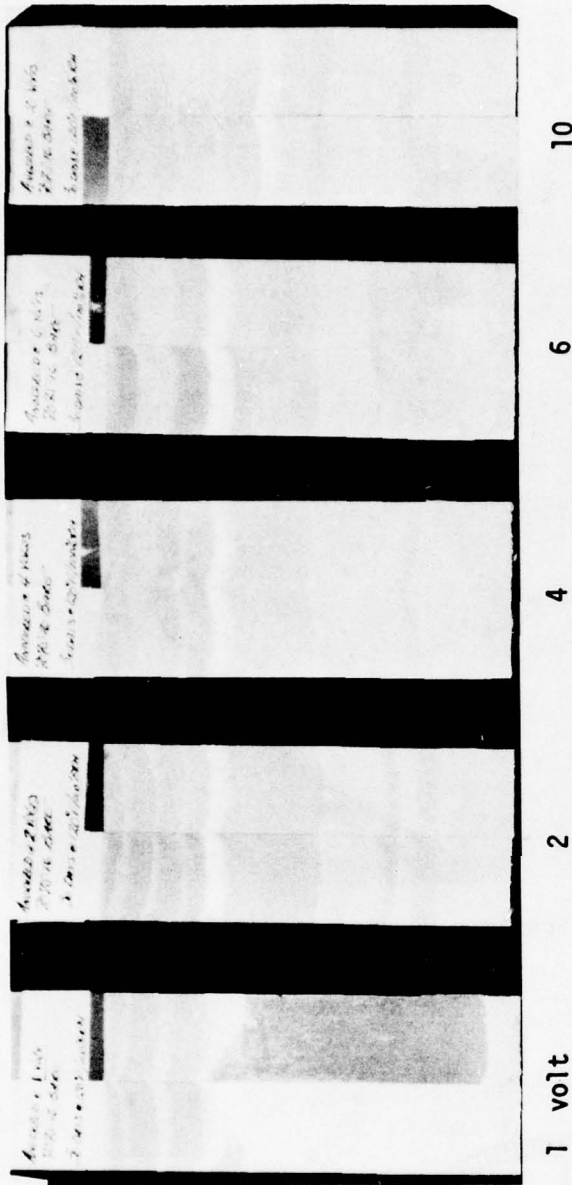


Figure 5 Typical Processing Sequence

7075-T6 Bare



2024-T3 Clad

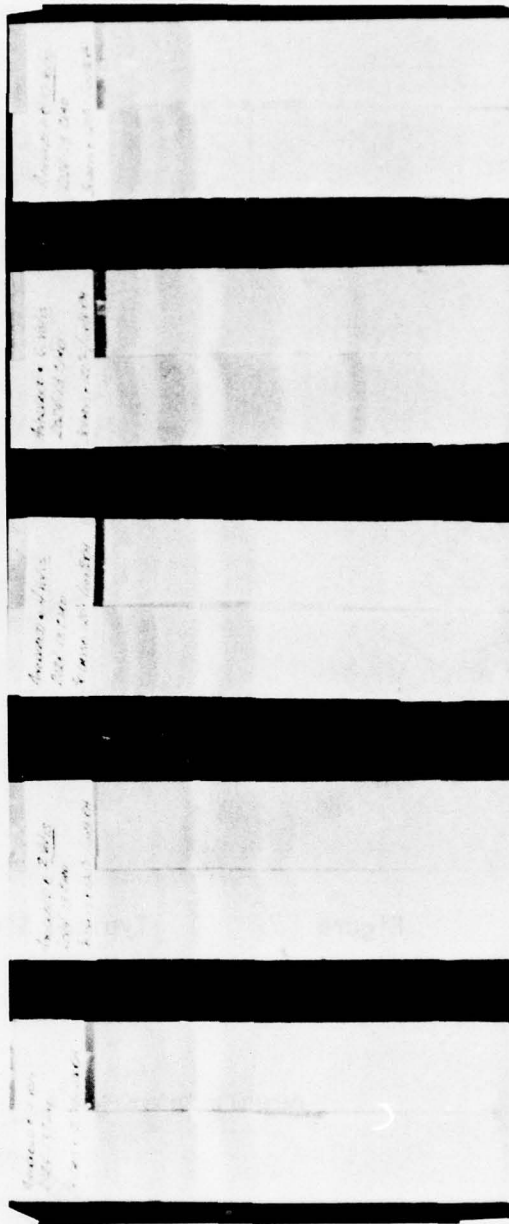


Figure 6 Wedge Test Specimens -- Exposed for 30 Days, 120°F/100% RH
Volatage Variation (FM 73/BR 127, PANTA)



Figure 7 Typical SEM Specimen Mounted on Viewing Stub

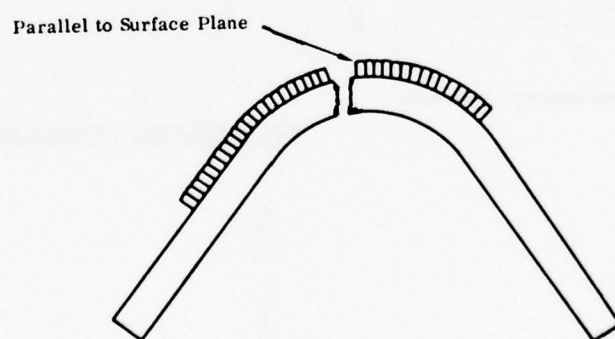


Figure 8 SEM Specimen Viewing Direction



Figure 9 SEM Photomicrograph of Oxide

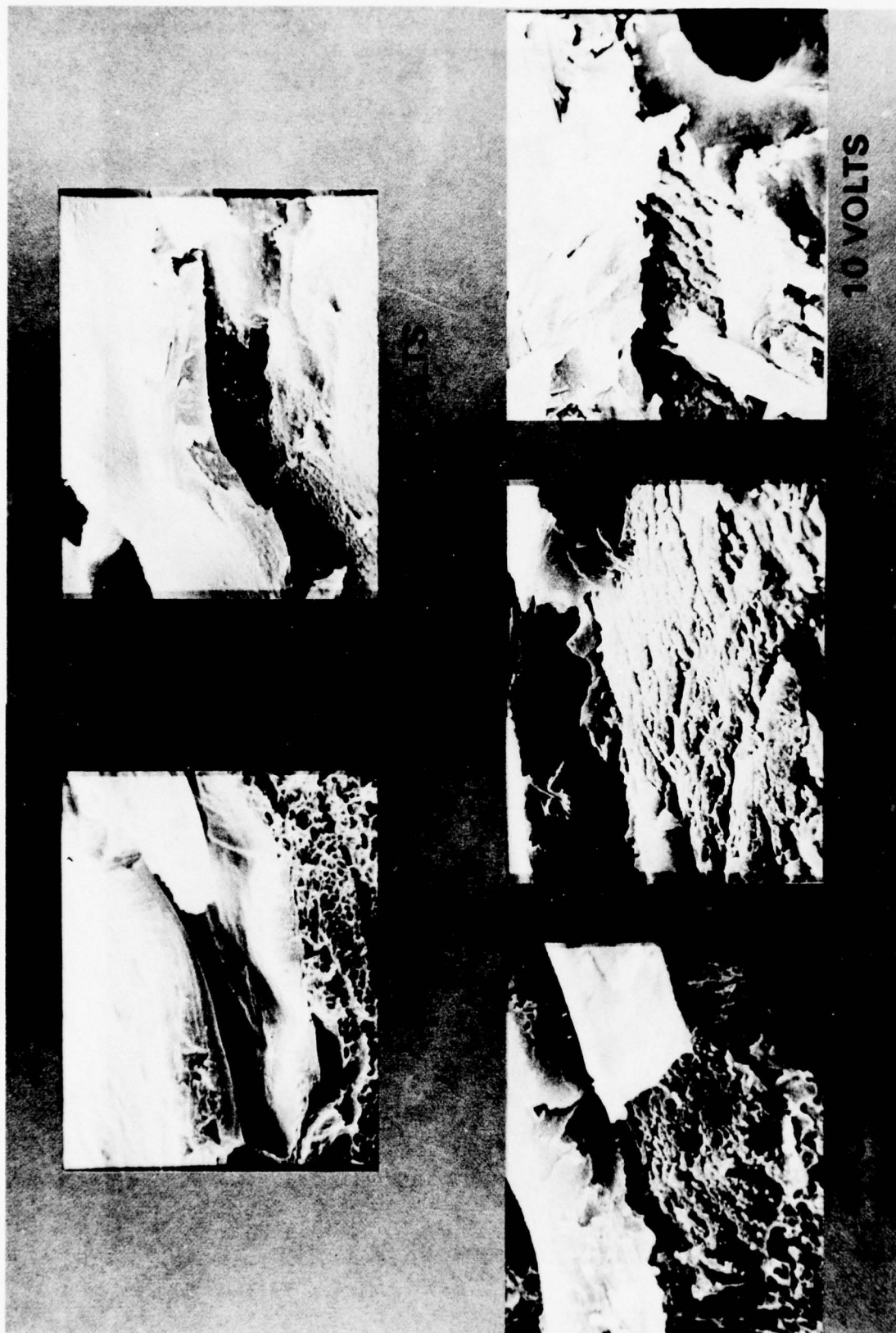


Figure 10 SEM Photomicrograph of Oxide as a Function of Voltage -- 7075-T6 Bare



Figure 11 SEM Photomicrograph of Oxide as a Function of Voltage -- 2024-T3 Clad

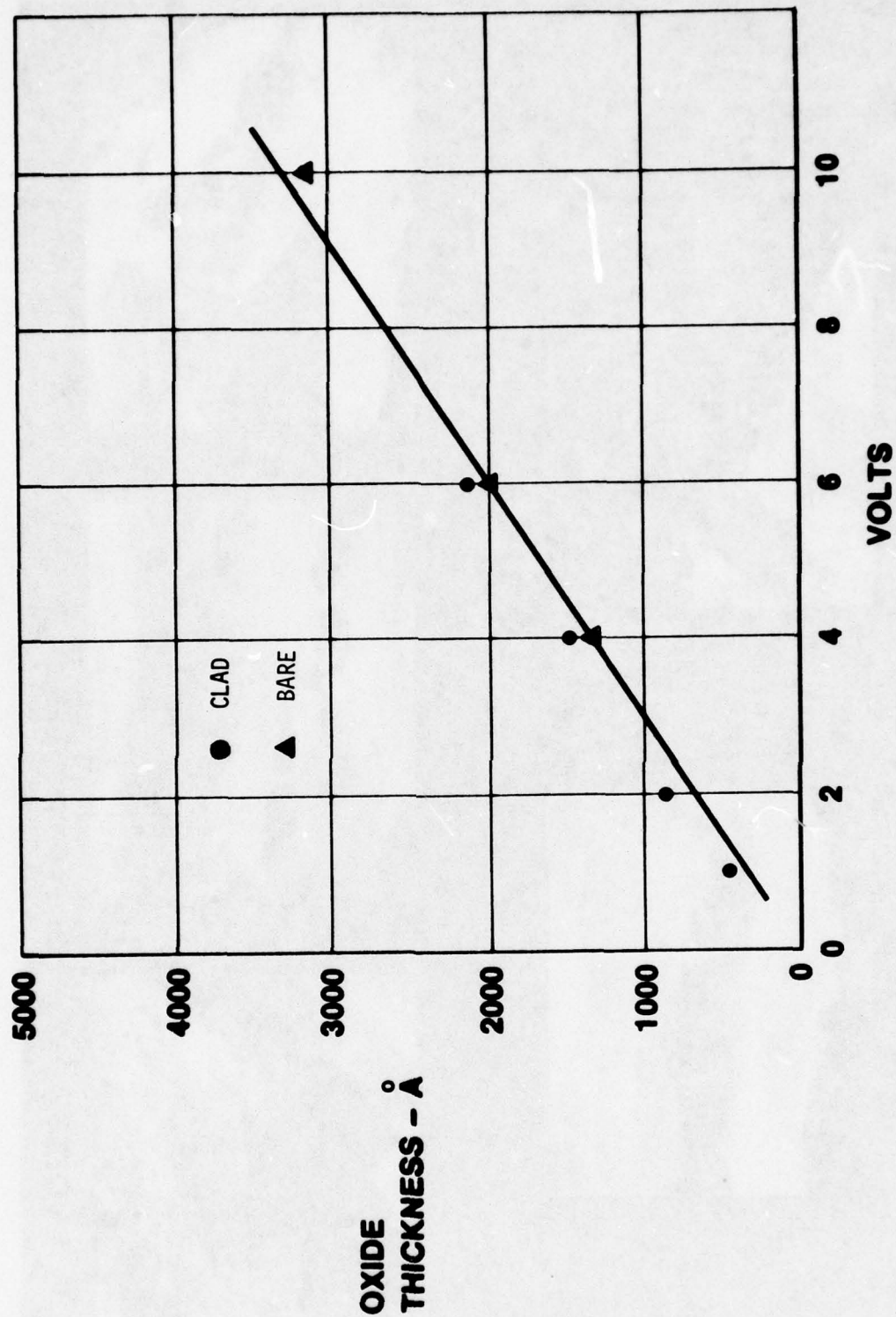


Figure 12 Oxide Thickness versus Voltage Variation

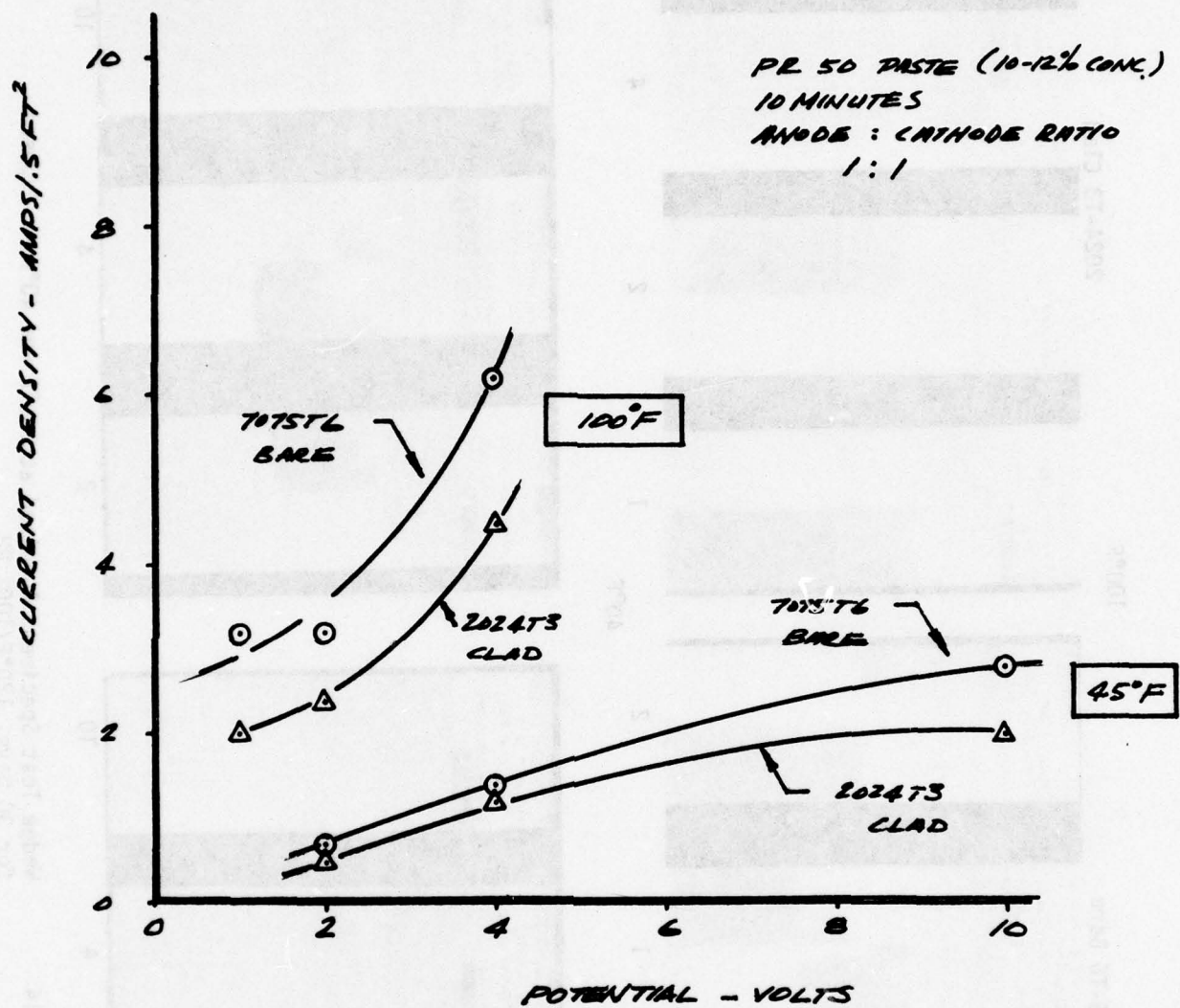


Figure 13 Effect of Temperature -- Voltage Variation versus Current Density

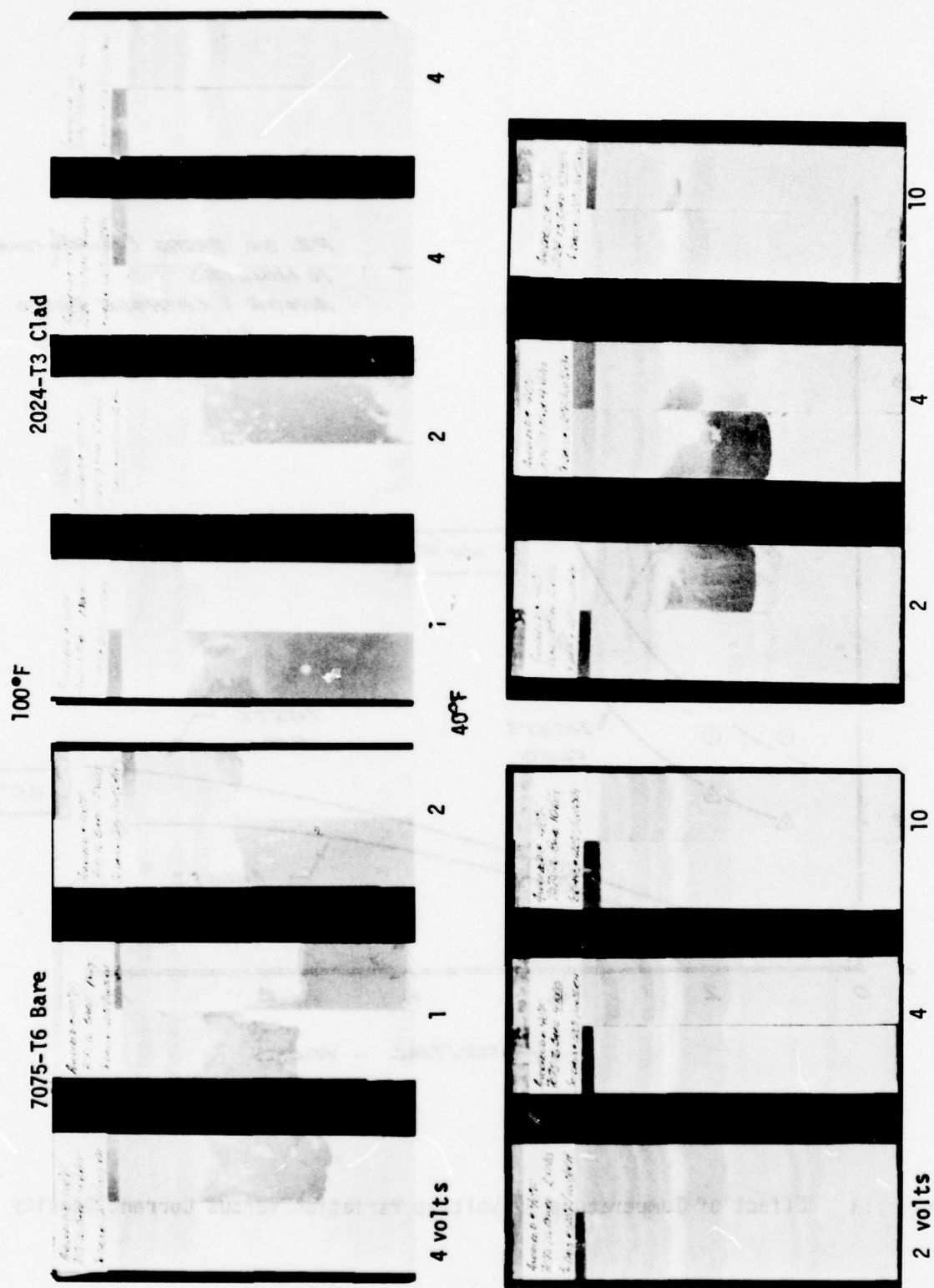


Figure 14 Wedge Test Specimens Anodized at 100°F and 40°F -- Exposed for 30 Days, 120°F/100% RH

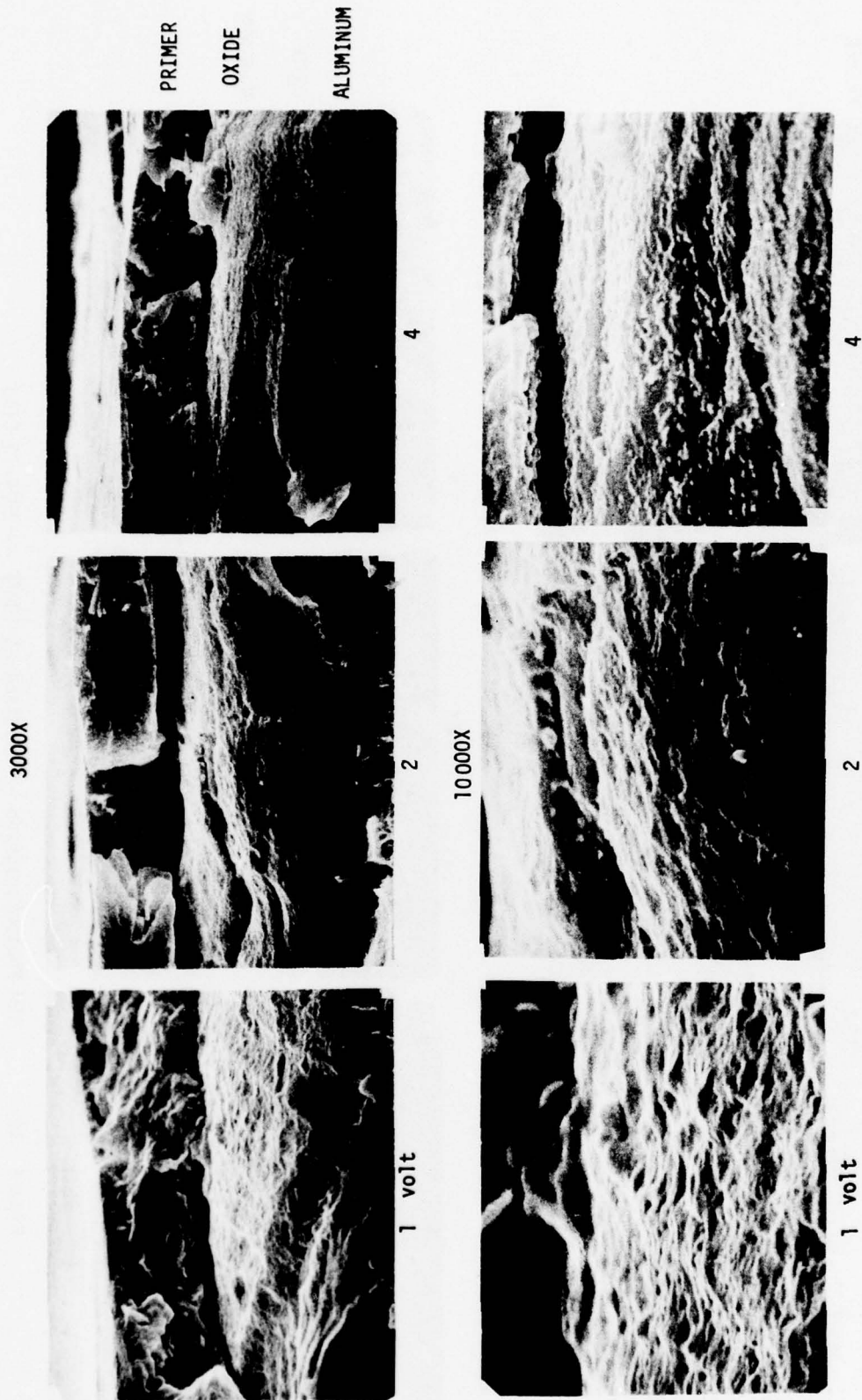


Figure 15 SEM Photomicrograph -- Anodized at 100°F -- 7075-T6 Bare

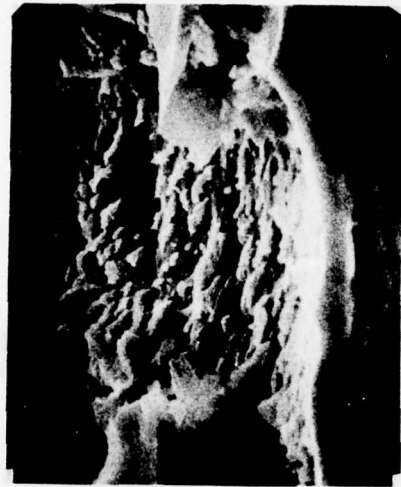
3000X



1 volt



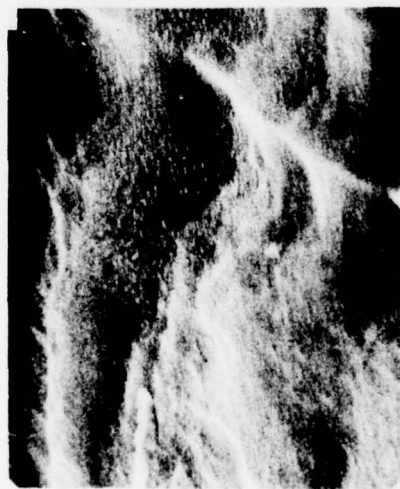
2



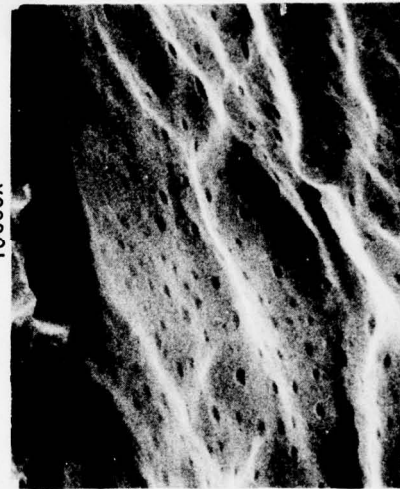
4

PRIMER

1000X



1 volt



2



4

OXIDE

ALUMINUM

Figure 16 SEM Photomicrograph -- Anodized at 100°F -- 2024-T3 Clad

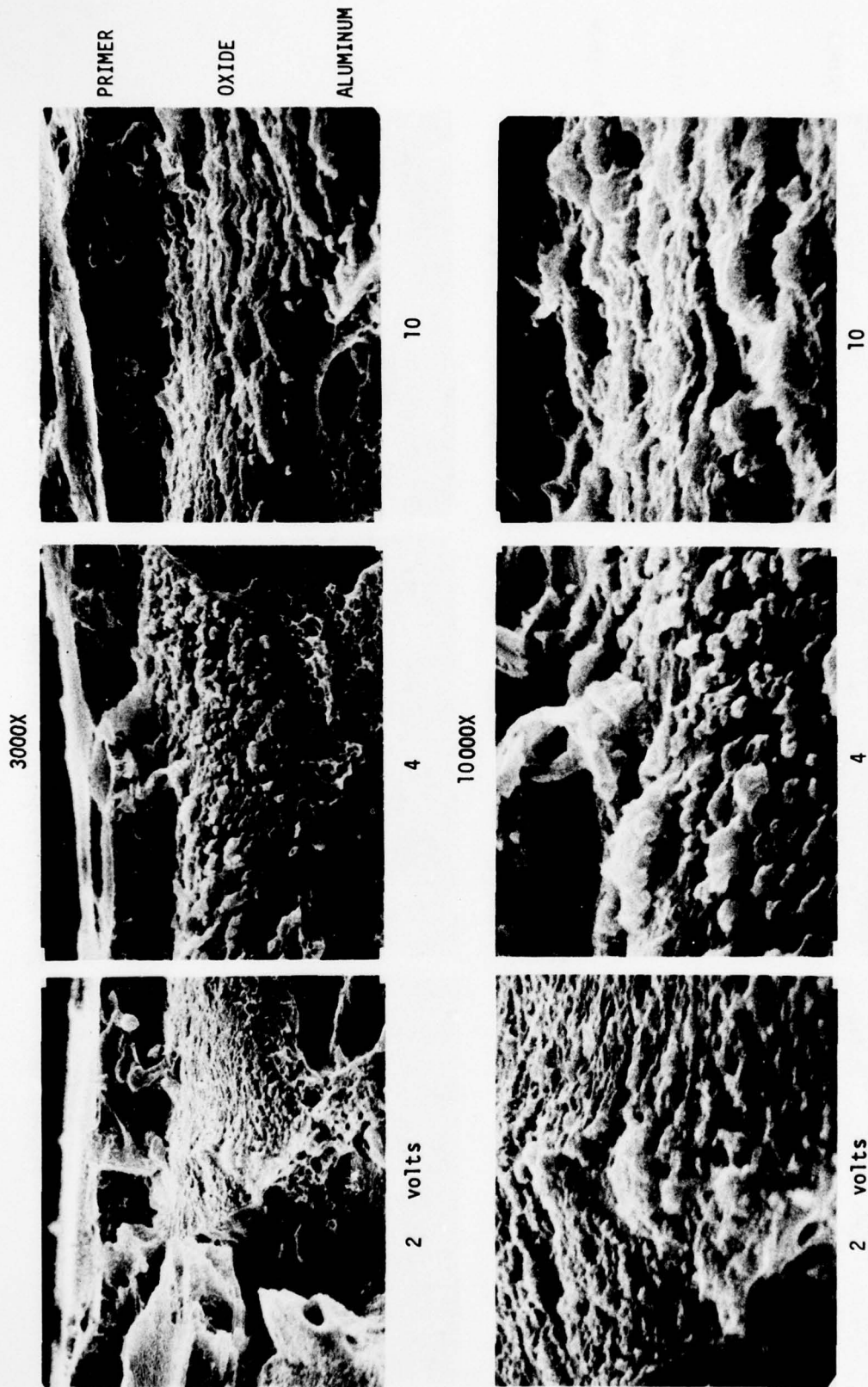


Figure 17 SEM Photomicrograph -- Anodized at 40°F -- 7075-T6 Bare

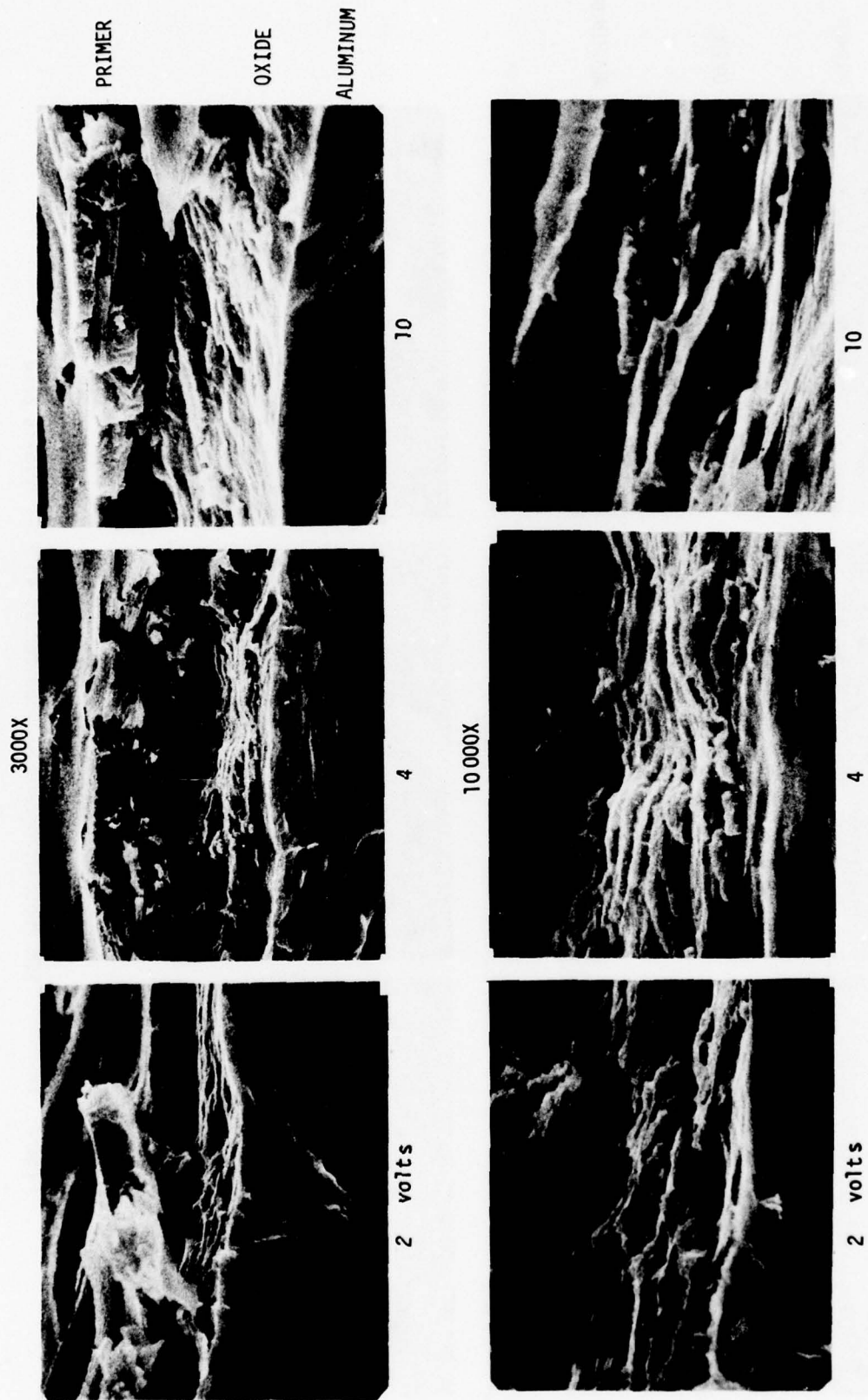
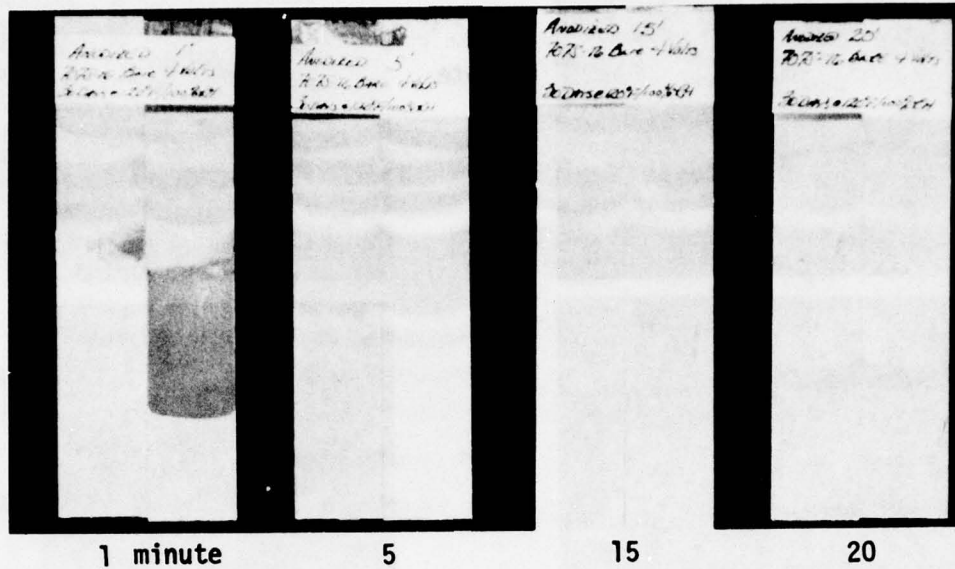


Figure 18 SEM Photomicrograph -- Anodized at 40°F -- 2024-T3 Clad

7075-T6 Bare



2024-T3 Clad

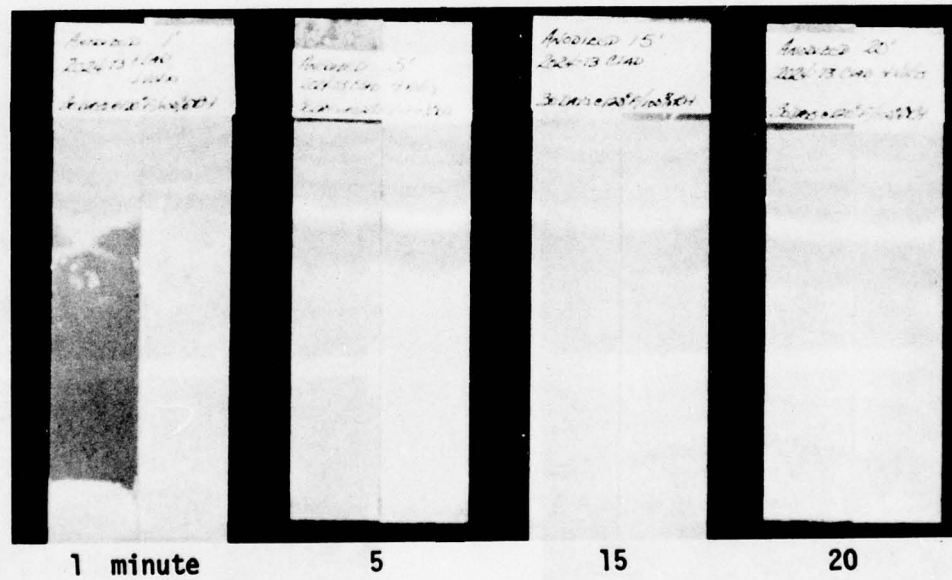
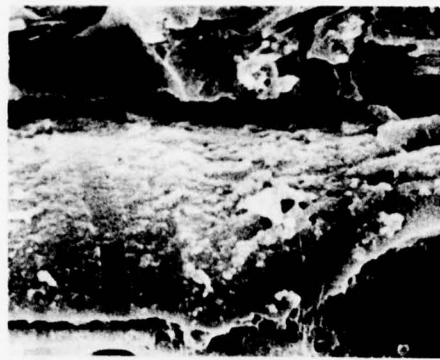
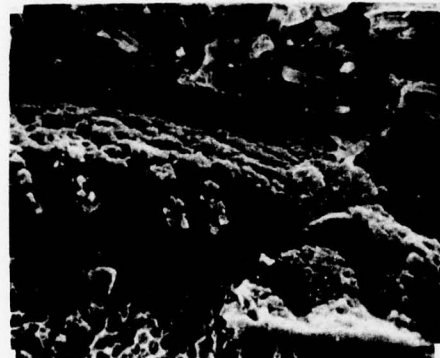


Figure 19 Effect of Anodize Time -- Wedge Test Specimens Exposed for 30 Days, 120°F/100% RH



1
minute



5



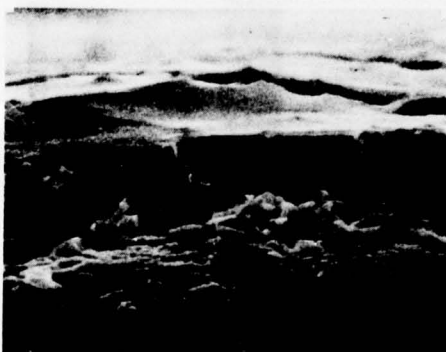
PRIMER

OXIDE

ALUMINUM



15



20



3000X

10 000X

Figure 20 SEM Photomicrograph -- Anodize Time Variation -- 7075-T6 Bare



1
minute



PRIMER

5
OXIDE

ALUMINUM



15



20

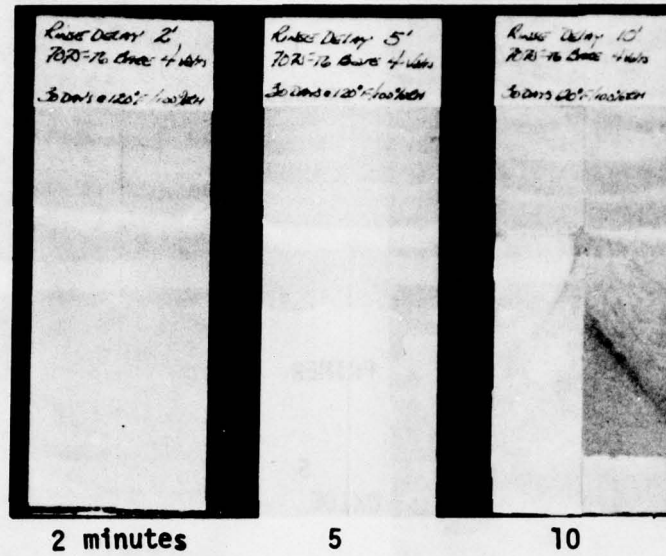


3000X

10000X

Figure 21 SEM Photomicrograph -- Anodize Time Variation -- 2024-T3 Clad

7075-T6 Bare



2024-T3 Clad

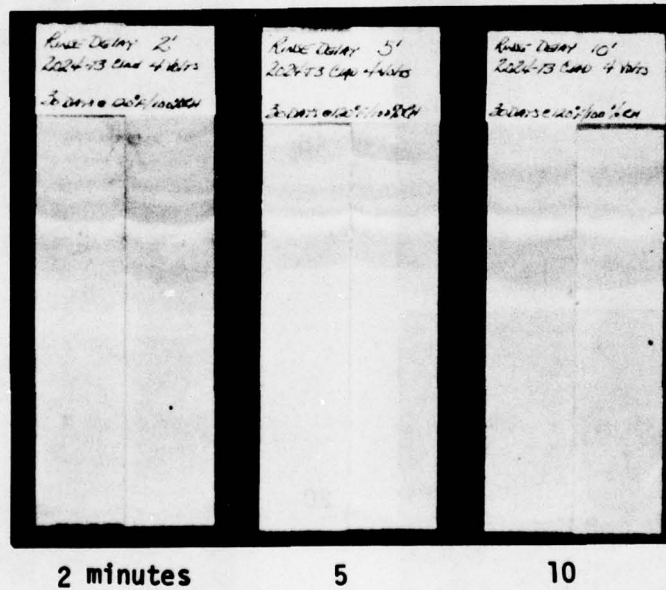
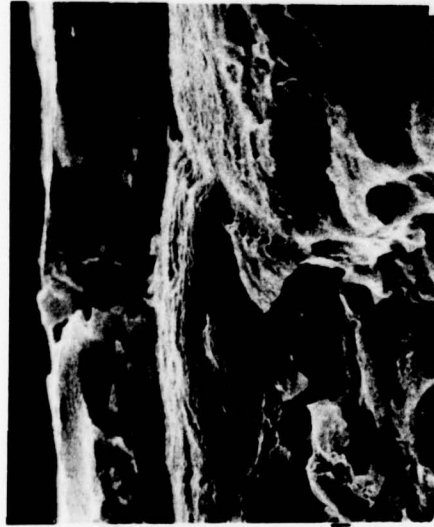


Figure 22 Effect of Rinse Delay Time -- Wedge Test Specimens Exposed
for 30 Days, 120°F/100% RH (FM 73/BR 127, PANTA)

3000X



PRIMER

OXIDE

ALUMINUM

2 minutes



5



10

10 000X



2 minutes

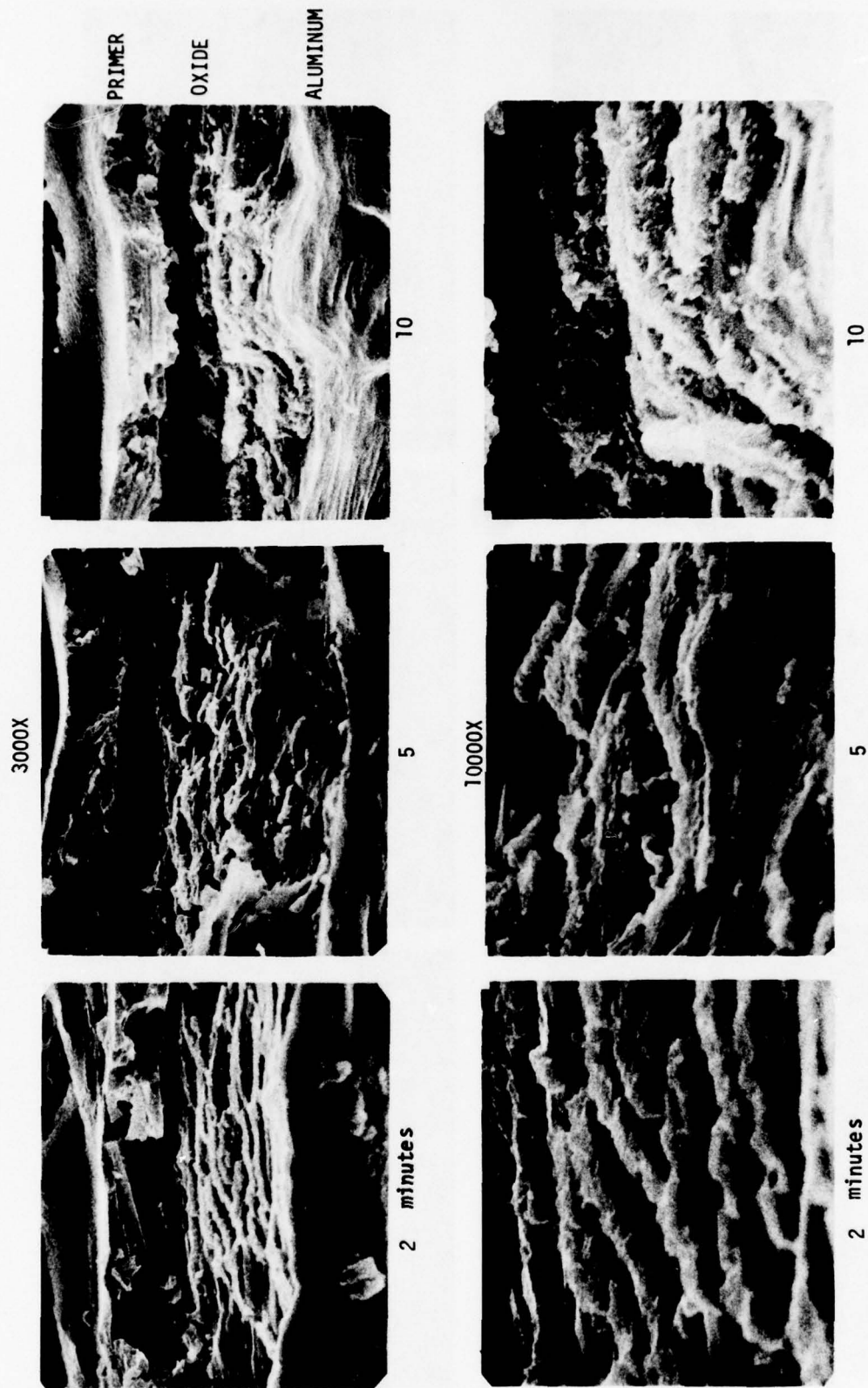


5



10

Figure 23 SEM Photomicrograph of Rinse Delay Time -- 7075-T6 Bare

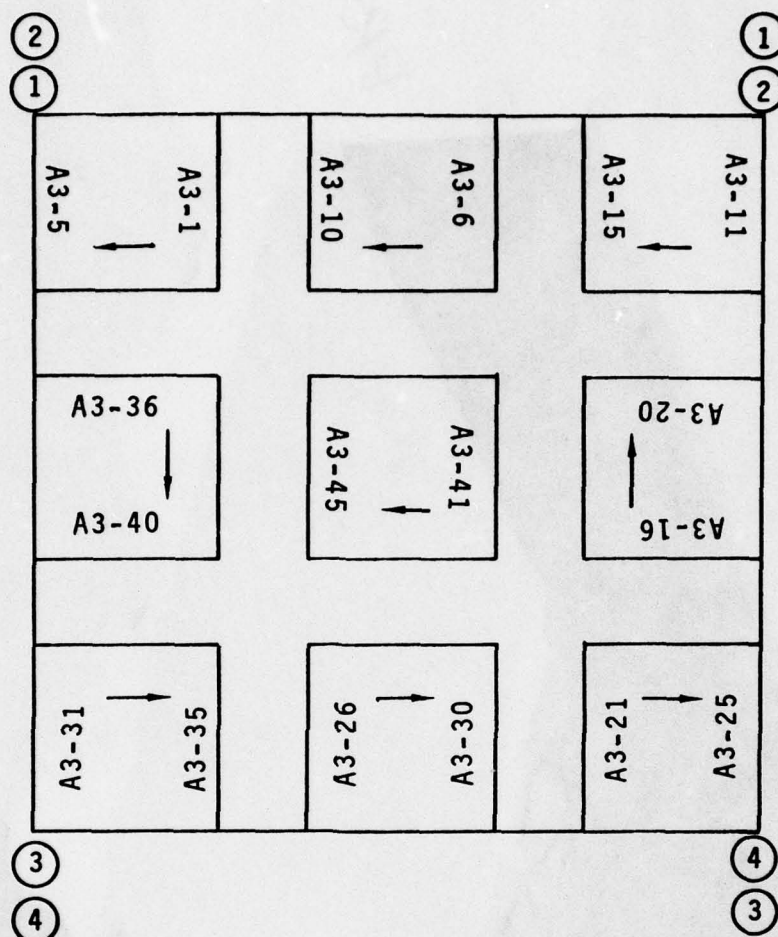


SEM Photomicrograph of Rinse Delay Time -- 2024-T3 Clad

Figure 24



Figure 25 Setup for Anodizing 2- x 2-ft Panel



NOTE: CIRCLED NUMBERS
ARE ELECTRICAL
CONTACTS

Figure 26

Panel Layout for Wedge Specimens

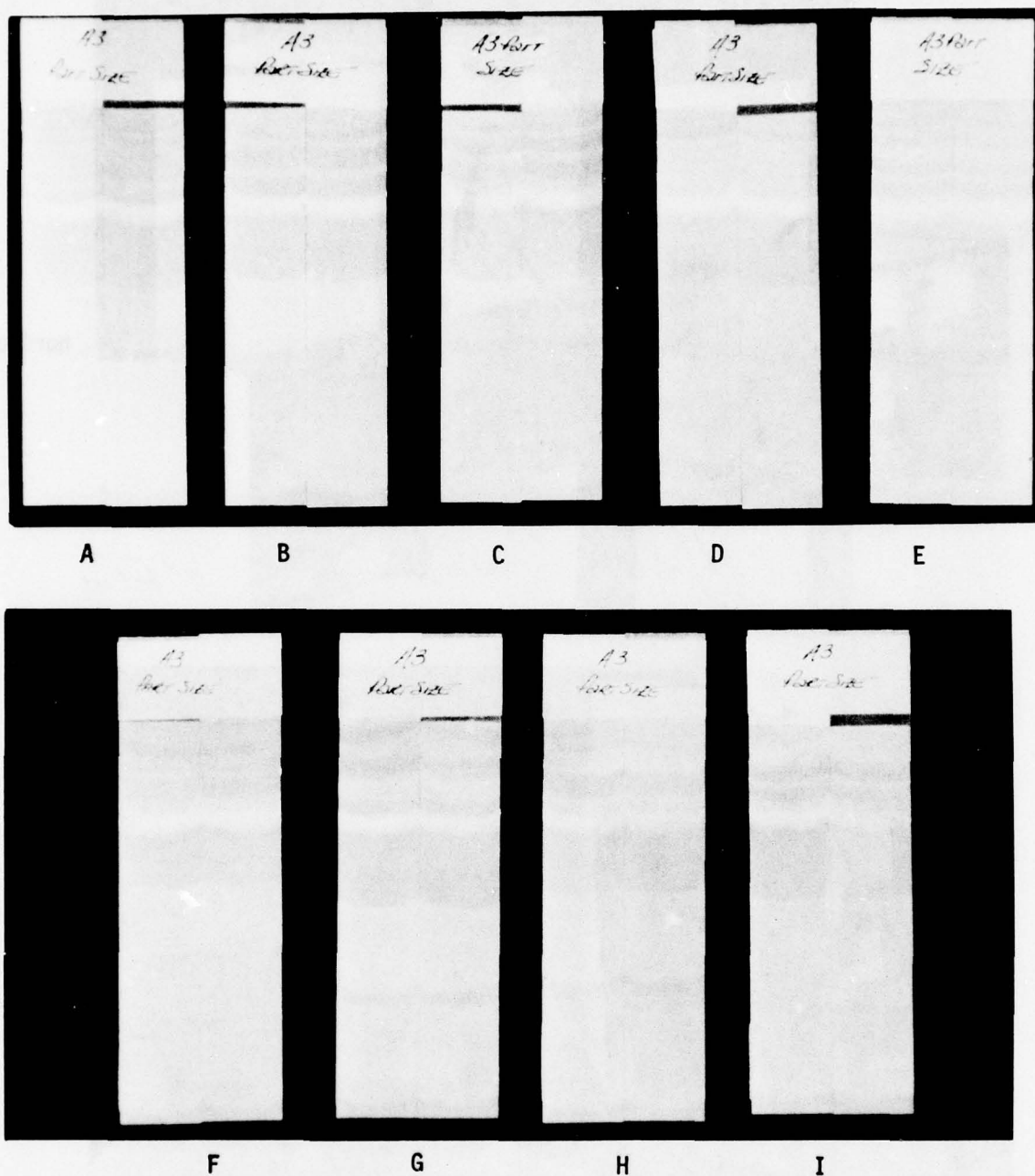
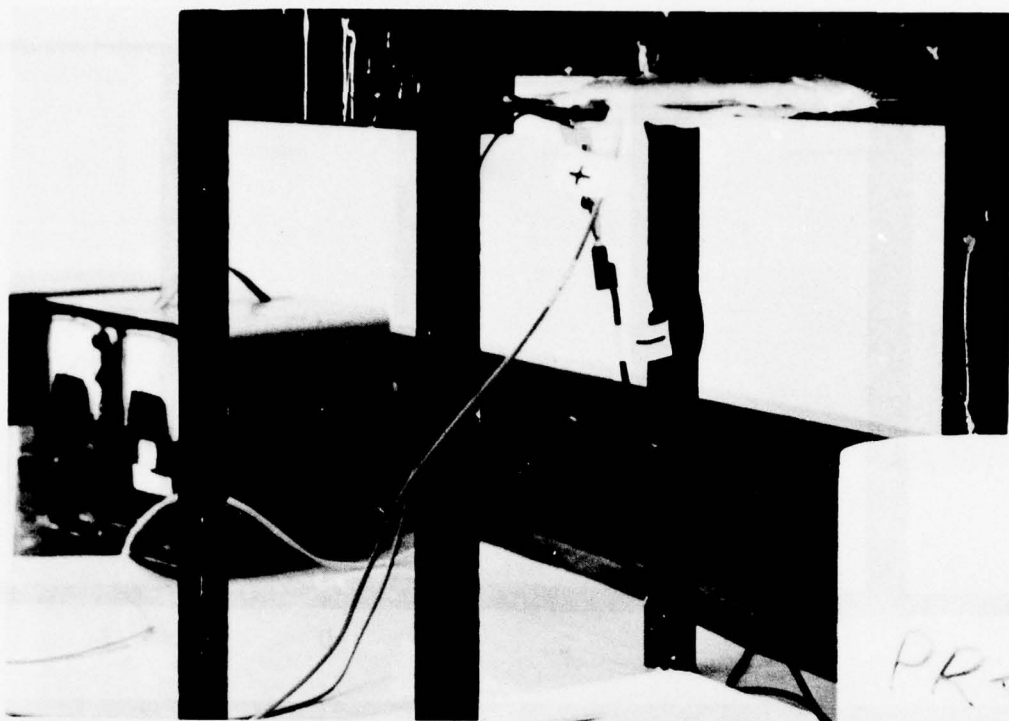
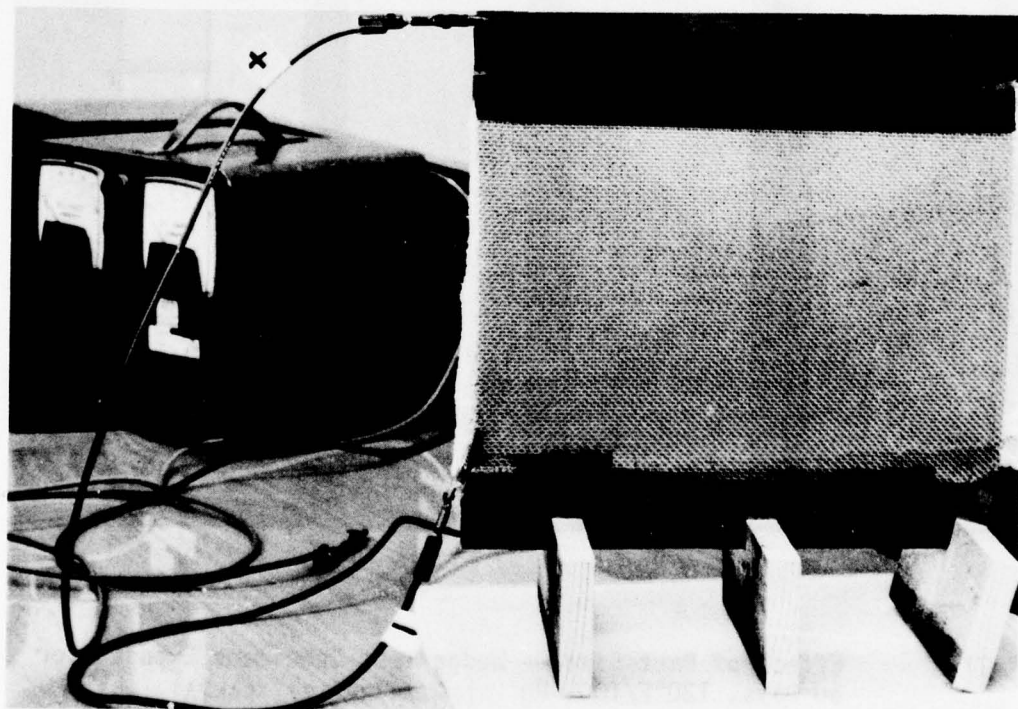


Figure 27

Effect of Part Size -- Wedge Test Specimens Exposed for
30 Days, 120°F/100% RH (FM 73/BR 127, PANTA)

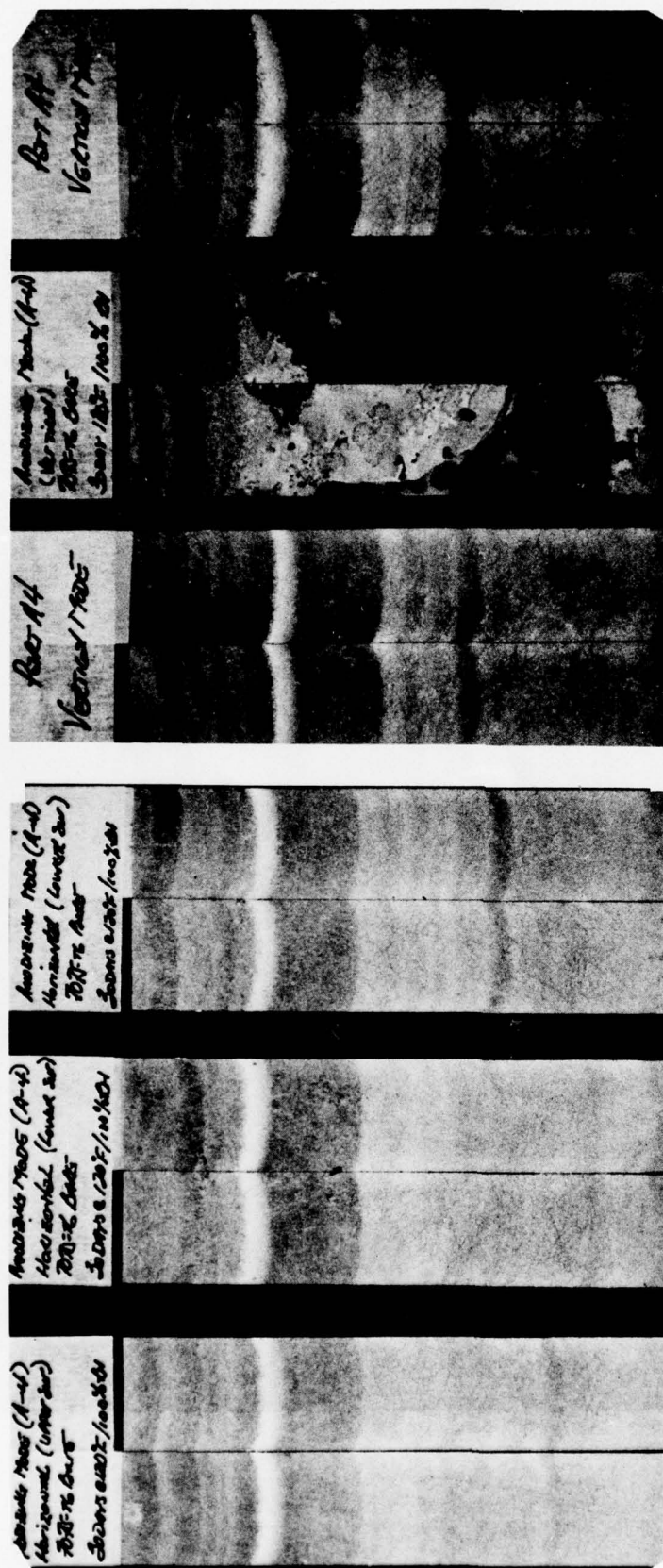


horizontal



vertical

Figure 28 Anodizing Mode Setup



horizontal
7075-T6 Bare

vertical
7075-T6 Bare

Figure 29 Effect of Anodizing Modes -- Wedge Test Specimens Exposed for 30 Days, 120°F/100% RH (FM 73/BR 127, PANTA)

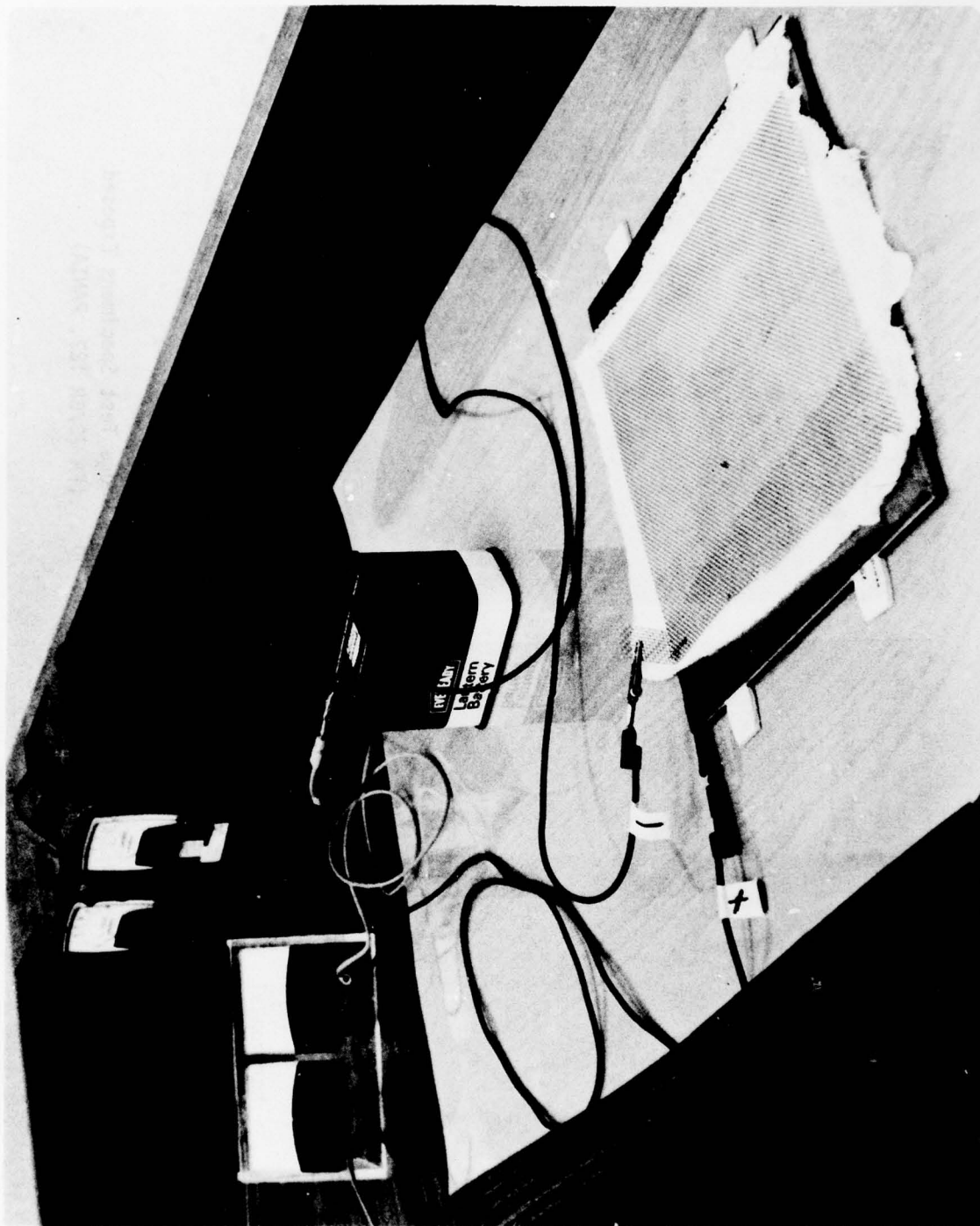


Figure 30 Battery Anodizing Setup (6 volt Battery)

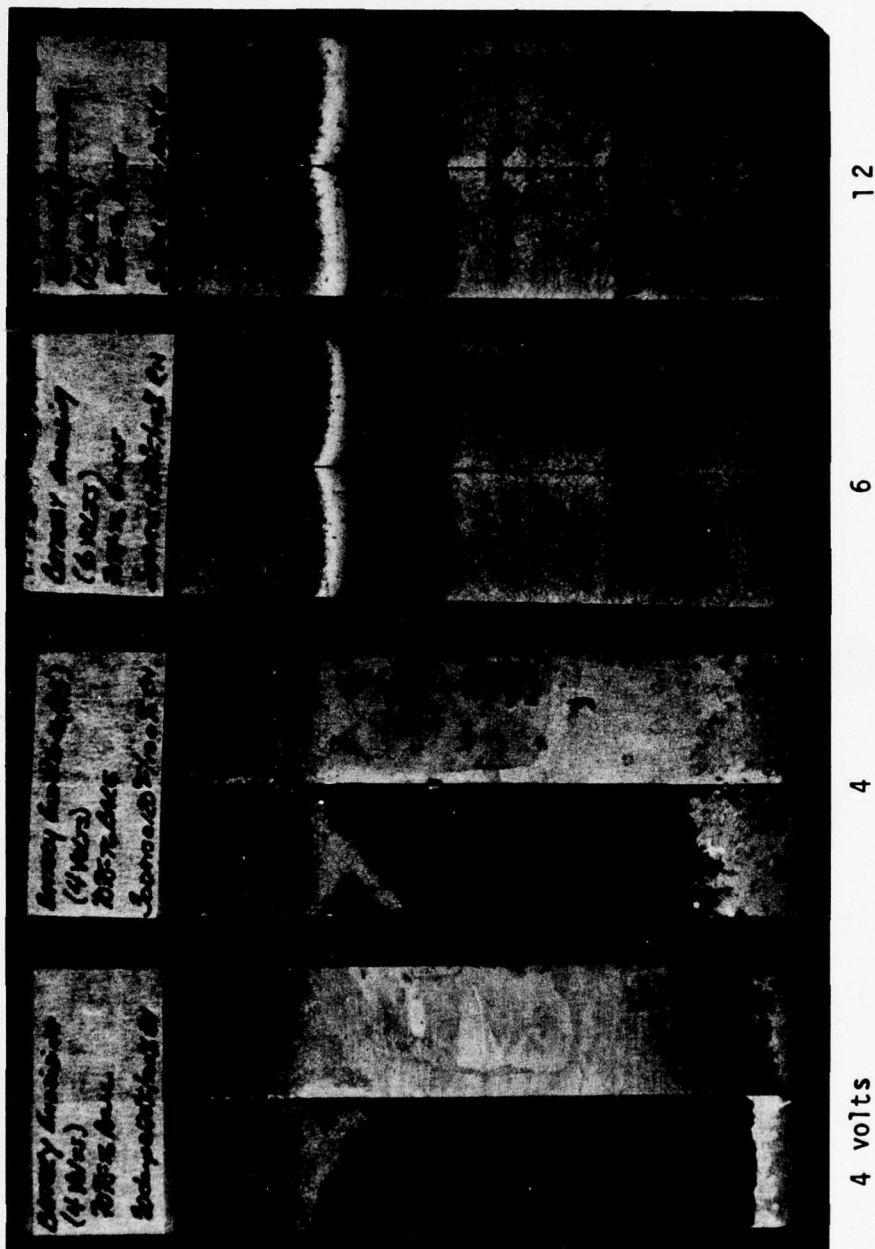
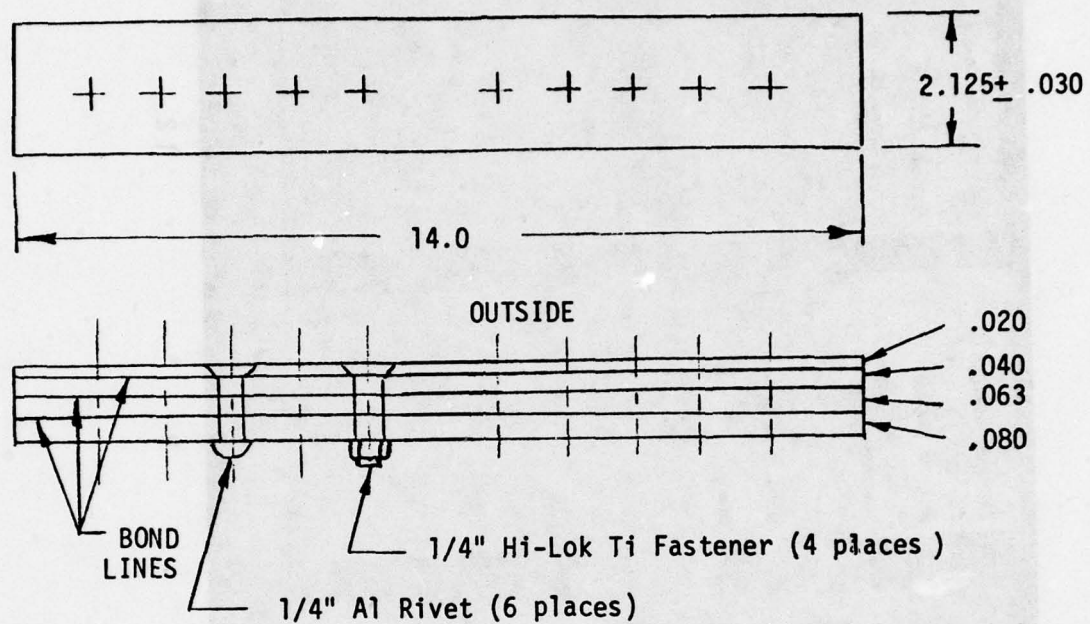


Figure 31 Battery Anodizing -- Wedge Test Specimens Exposed for 30 Days, 120°F/100% RH (FM 73/BR 127, PANTA) 7075-T6 Bare



BONDING SYSTEM

- .7075T6 Bare
- .FM73/BR127
- .Phosphoric Acid Anodize BAC 5555

Figure 32 Fastener Specimen

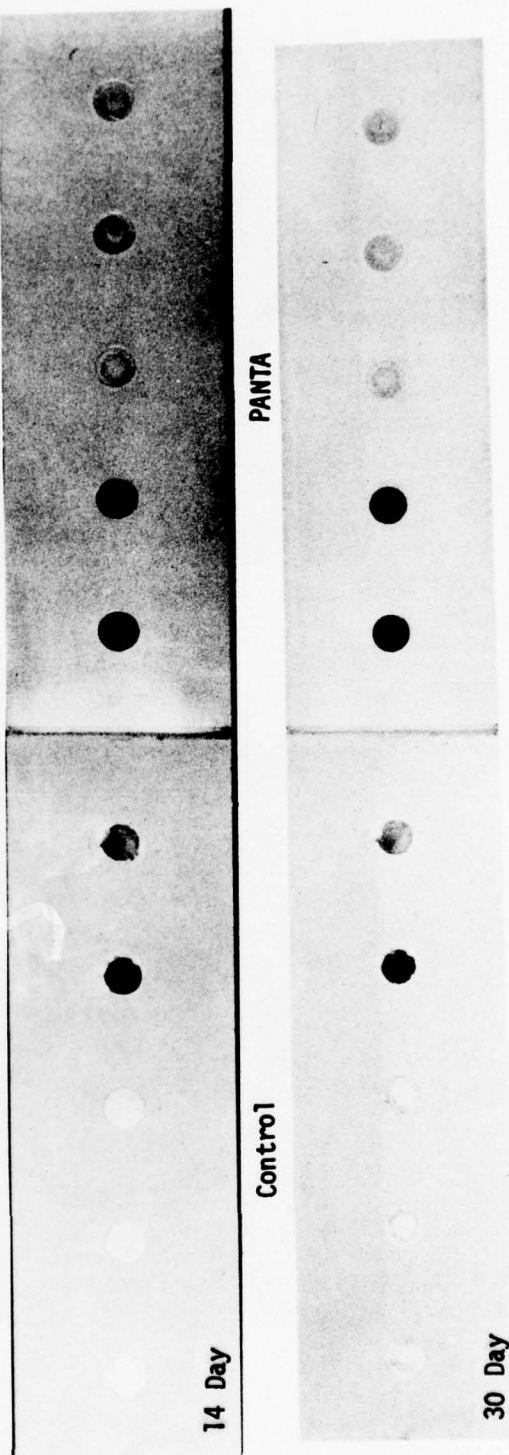
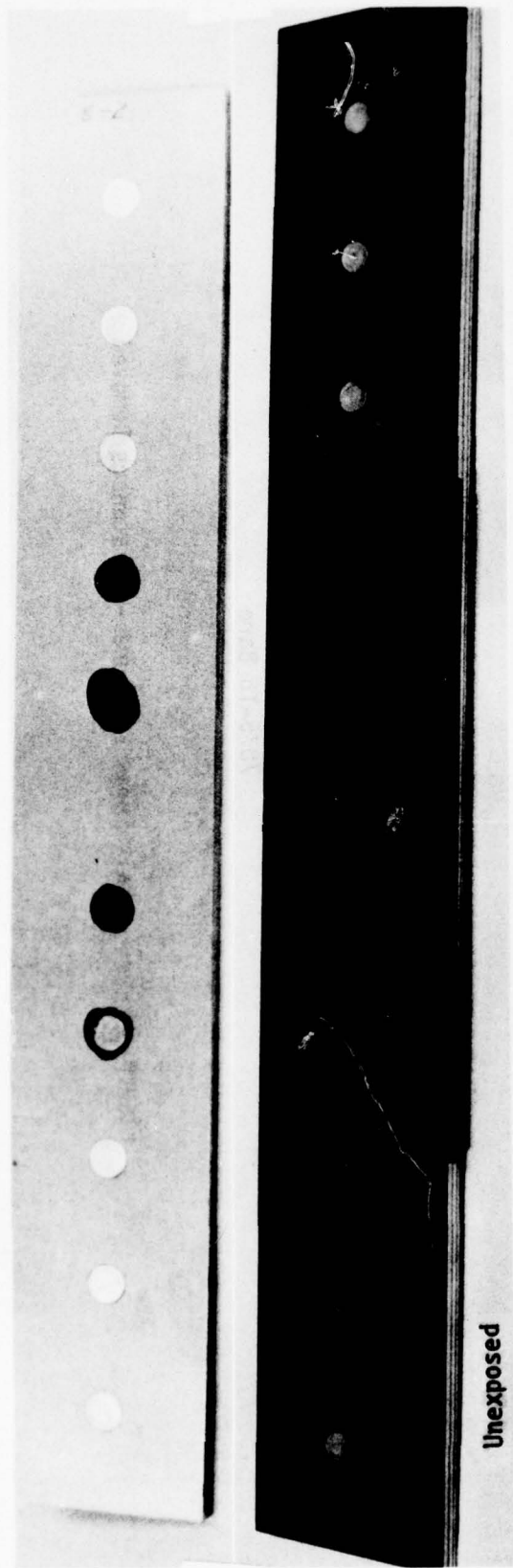


Figure 33 Fastener Specimen -- Control versus Salt Spray Exposure 7075-T6 Bare



Control



PANTA

FM73/BR127
7075-T6 Bare

Figure 34 Salt Spray Exposure -- Fasteners Removed

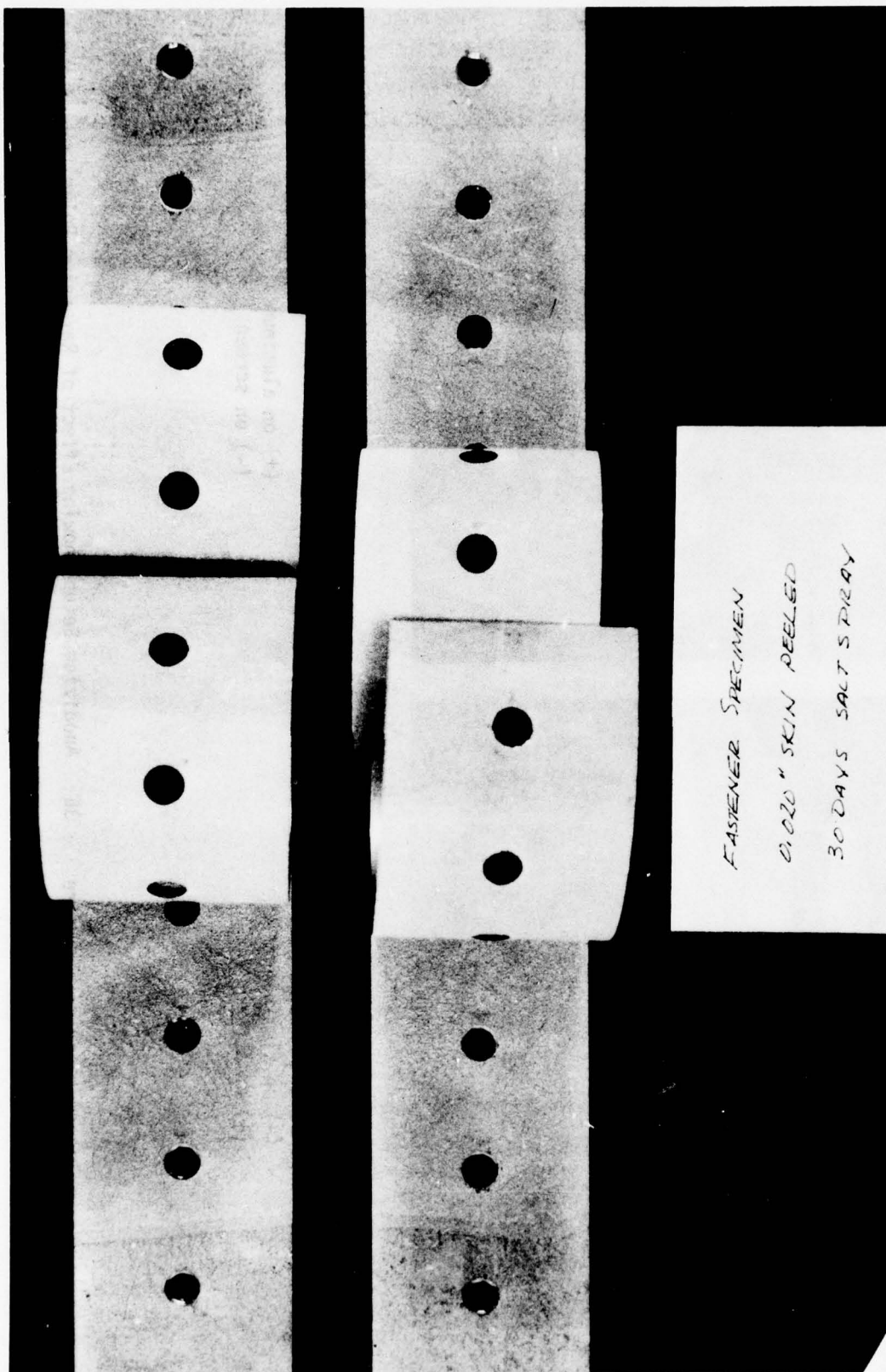
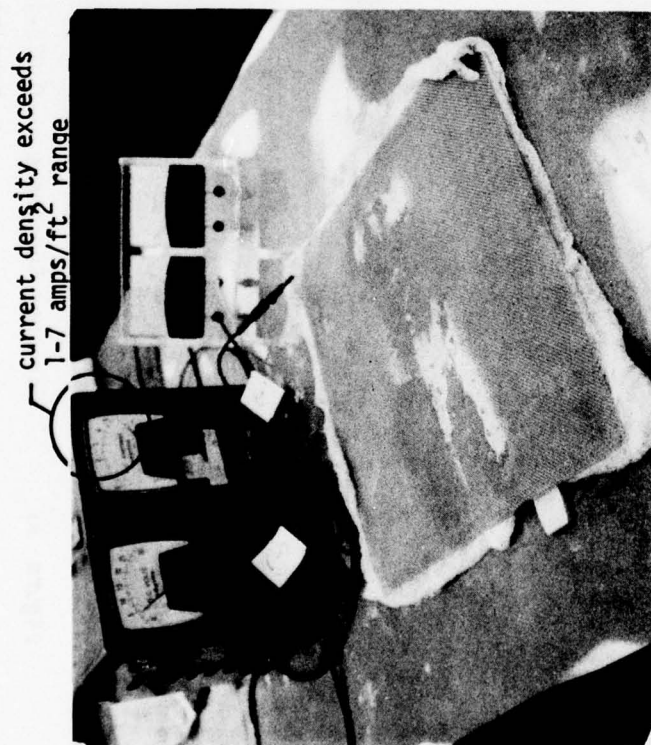


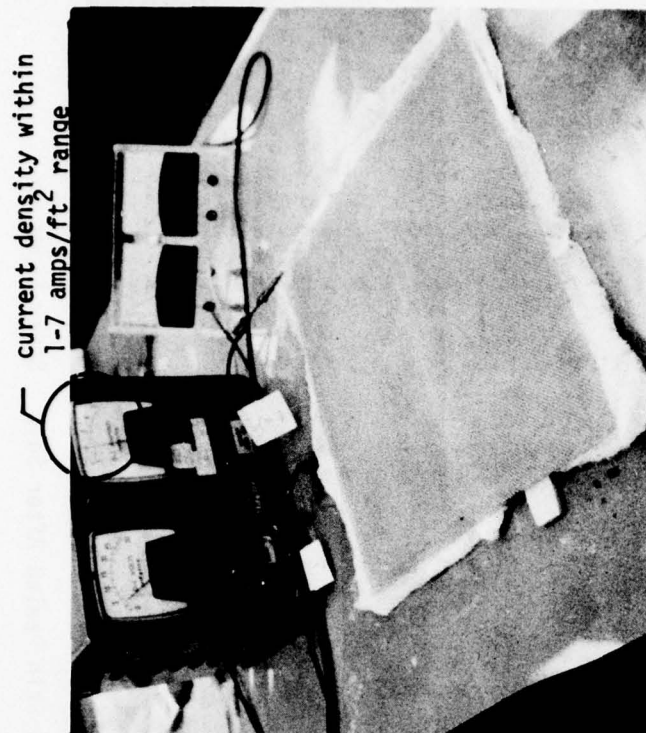
Figure 35 Fastener Specimens -- 0.020-inch Skin Peeled After Salt Spray Exposure
7075-T6 Bare



Reverse Leads

Incorrect Procedure

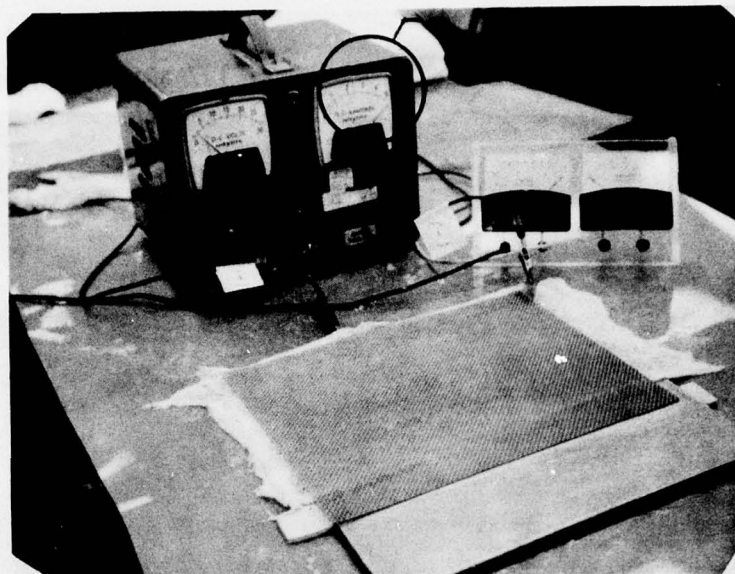
- (-) on aluminum
- (+) on screen



Correct Procedure

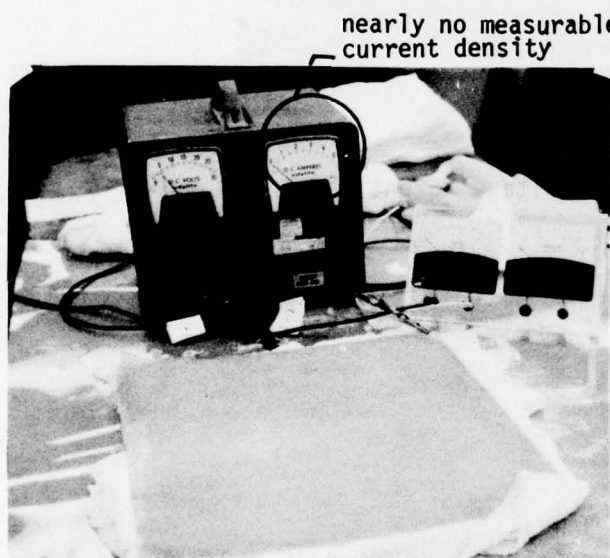
- (+) on aluminum
- (-) on screen

Figure 36 Anodizing Setup Showing Effect of Reversing Polarity

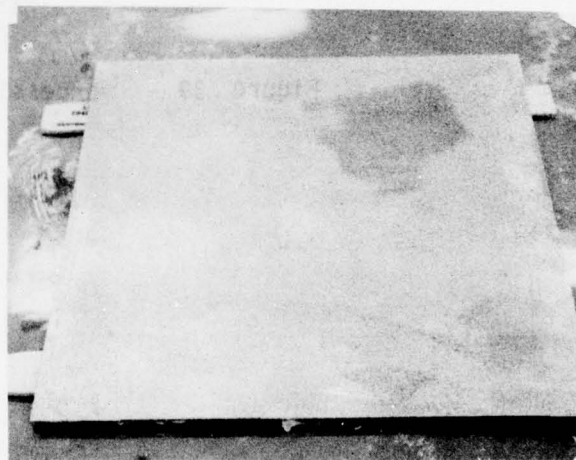


current density
exceeds
1-7 amps/ft² range

Figure 37 Screen Contacting Aluminum



nearly no measurable
current density

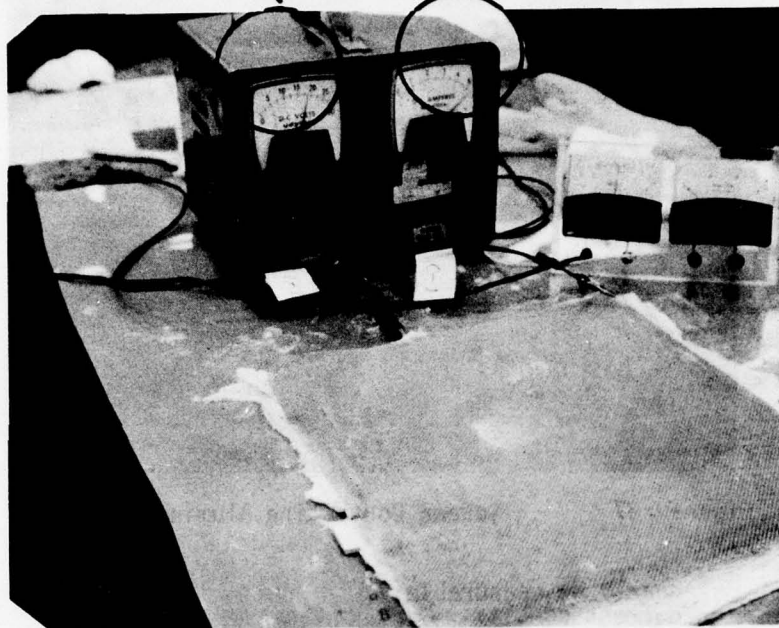


Resulting Surface

Figure 38 Anodizing Showing Effect of Inadequate Paste

voltage potential
exceeds 10 volts

current density exceeds
1-7 amps/ft² range



Over 10 Volts

Figure 39 Excessive Voltage Potential

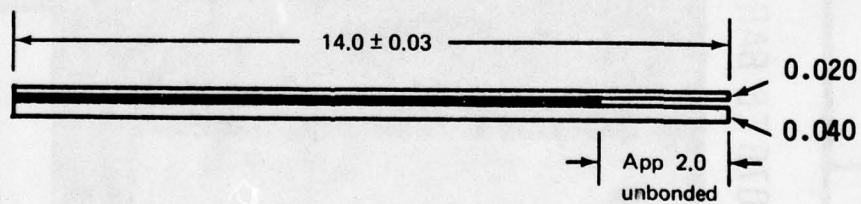
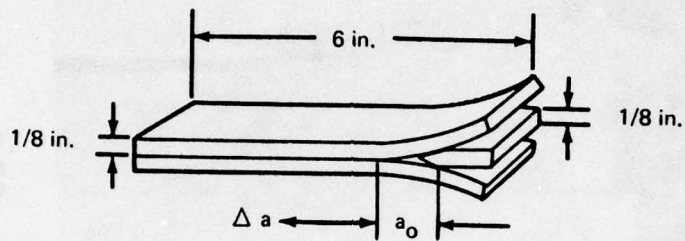
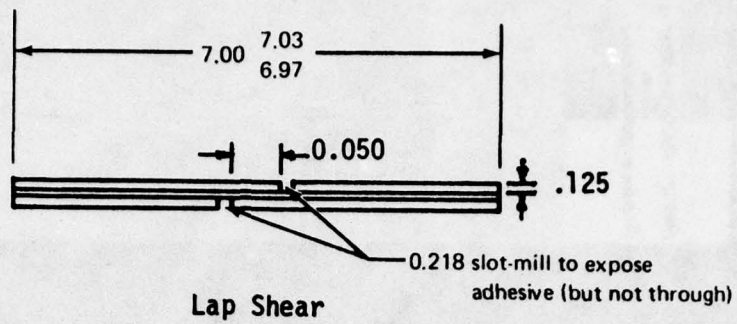


Figure 40 Specimen Configurations

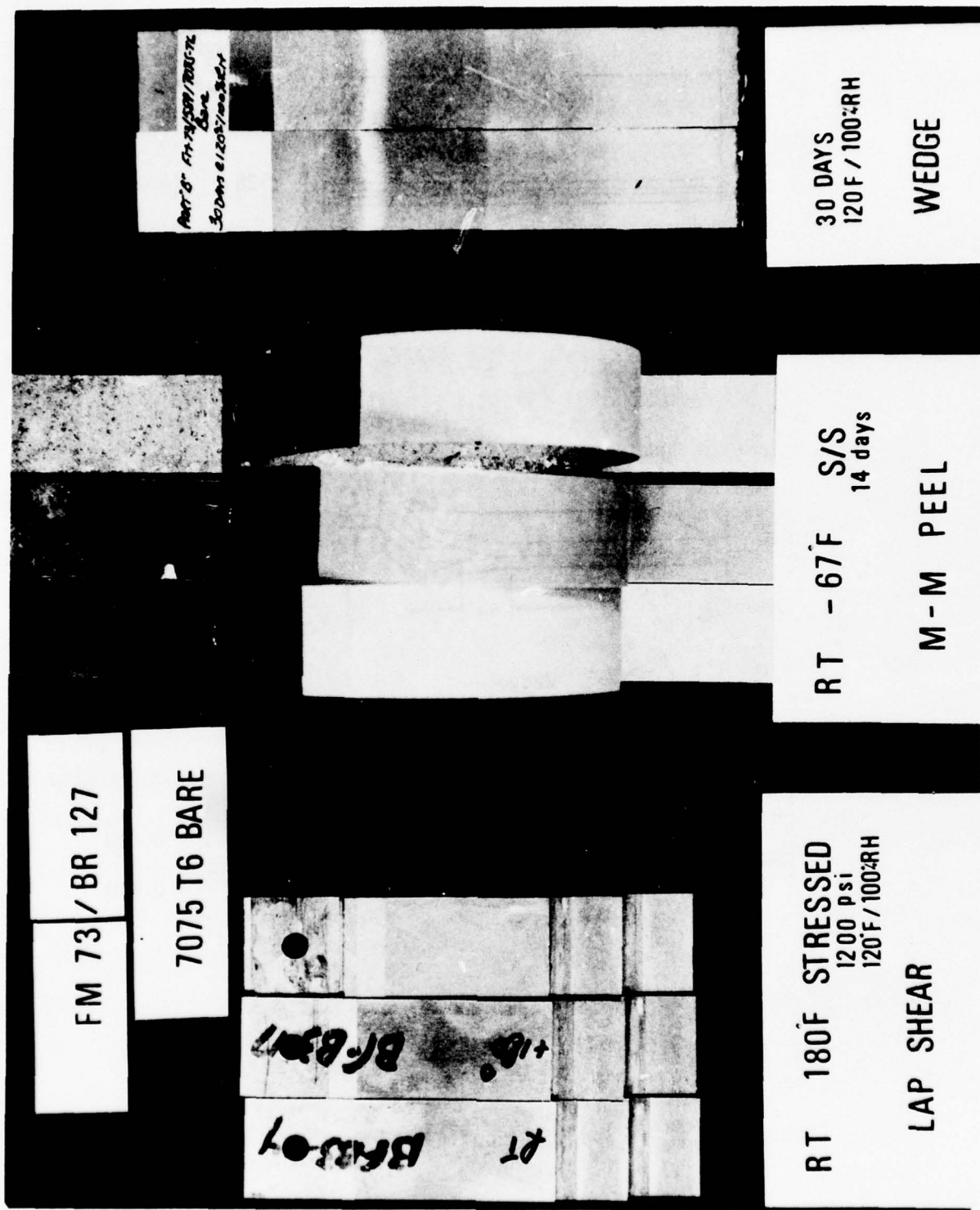


Figure 41 Specimen Bond Surfaces -- FM 73, 7075-T6 Bare

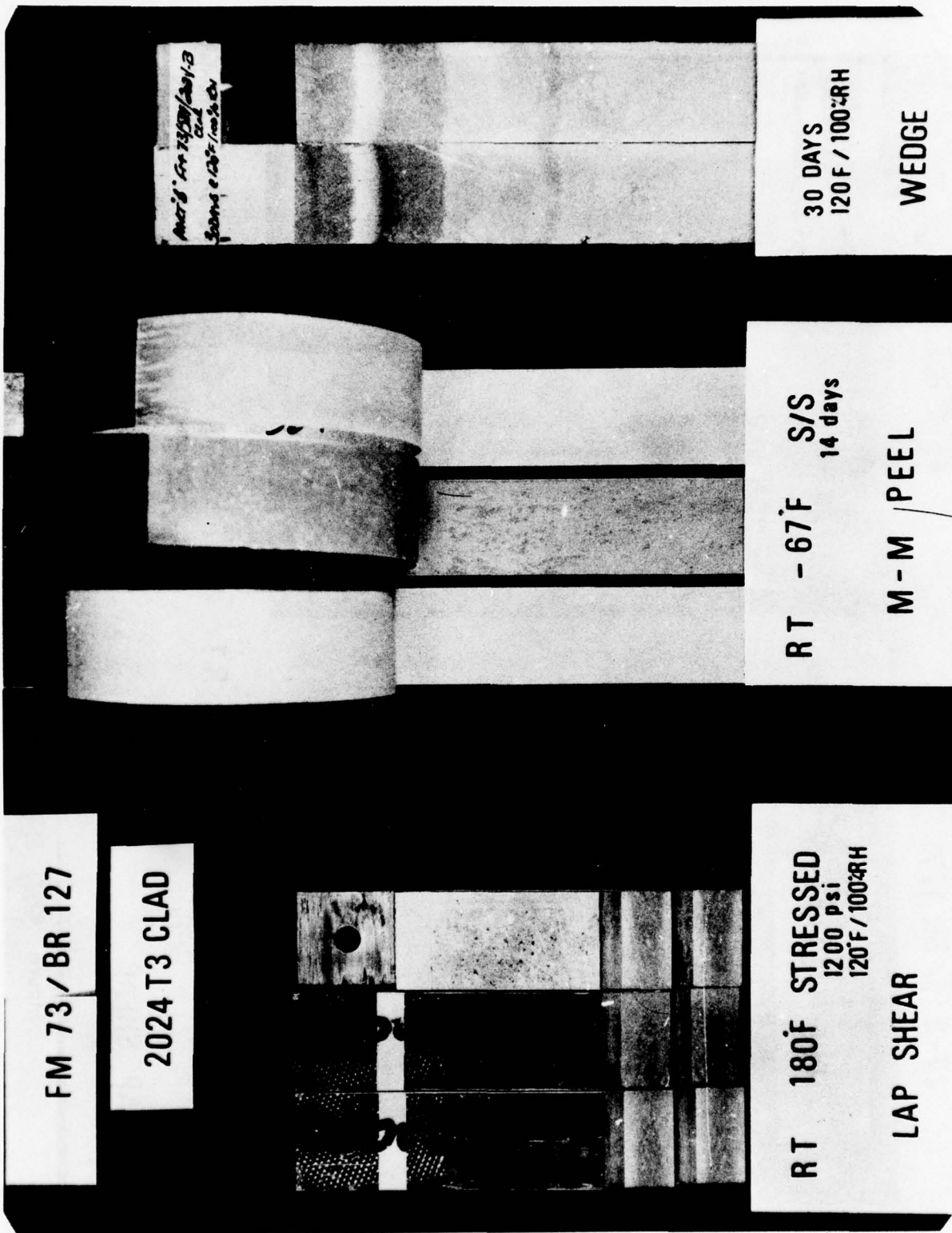


Figure 42 Specimen Bond Surfaces -- FM 73, 2024-T3 Clad

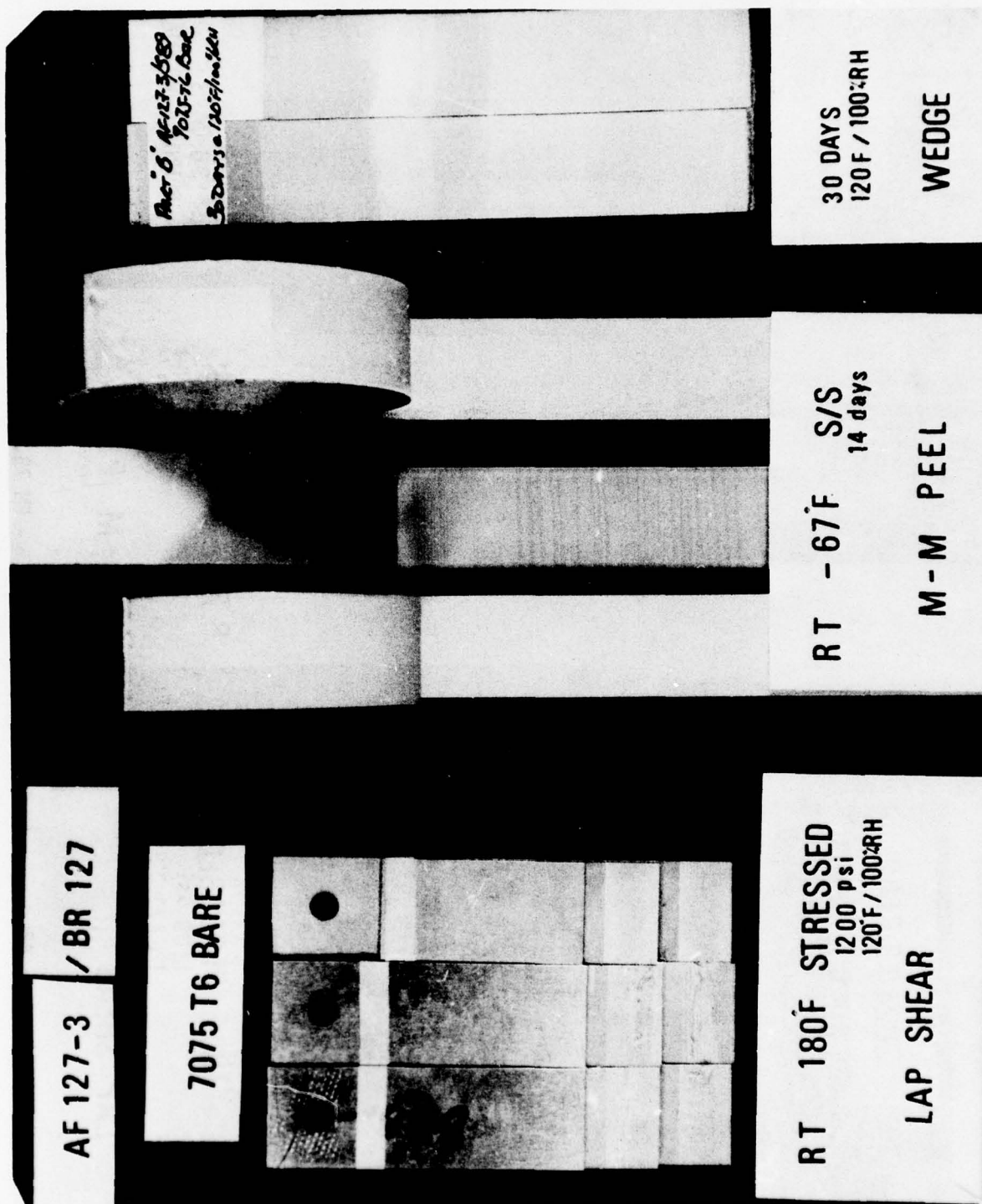


Figure 43 Specimen Bond Surfaces -- AF 127-3, 7075-T6 Bare

AF 127-3 / BR 127

2024 T3 CLAD

Aug 18th 4:17:33 / 588
2024-78 clad
30 days @ 120°F / 100%RH

RT 180°F STRESSED
1200 psi
120°F / 100%RH

LAP SHEAR

RT - 67°F S/S
14 days

M - M PEEL

30 DAYS
120°F / 100%RH

WEDGE

Figure 44 Specimen Bond Surfaces -- AF 127-3, 2024-T3 Clad

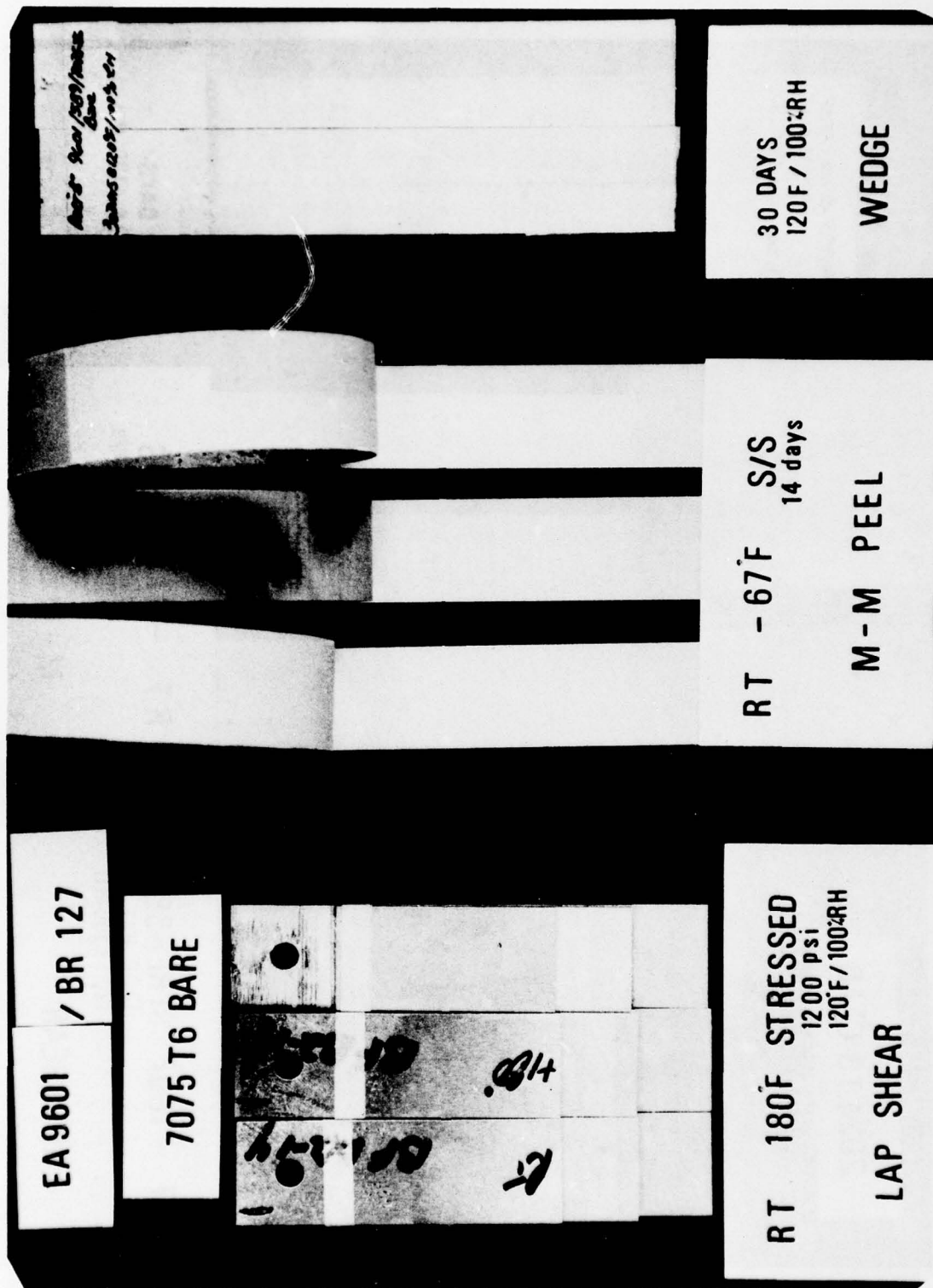


Figure 45 Specimen Bond Surfaces -- EA 9601, 7075-T6 Bare

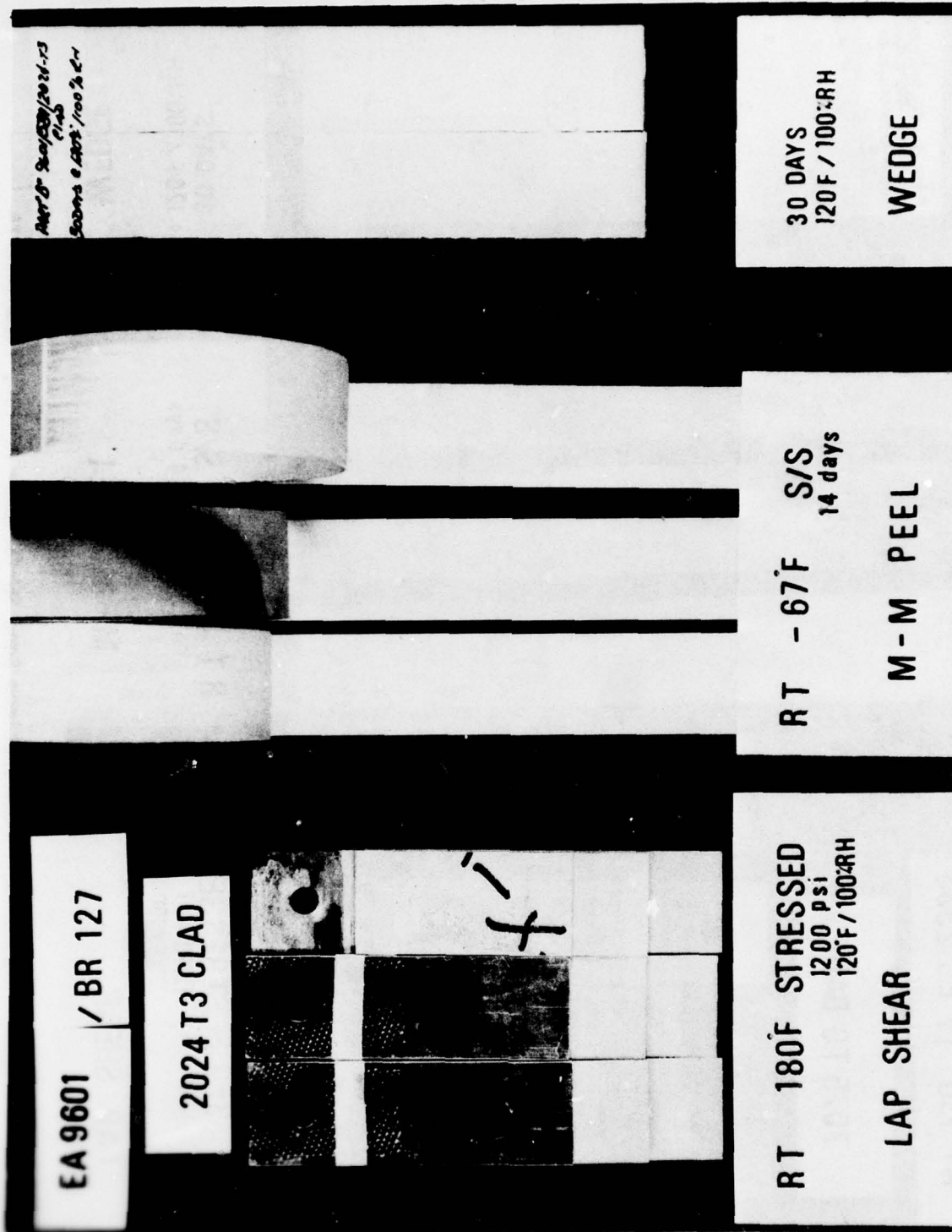


Figure 46 Specimen Bond Surfaces -- EA 9601, 2024-T3 Clad

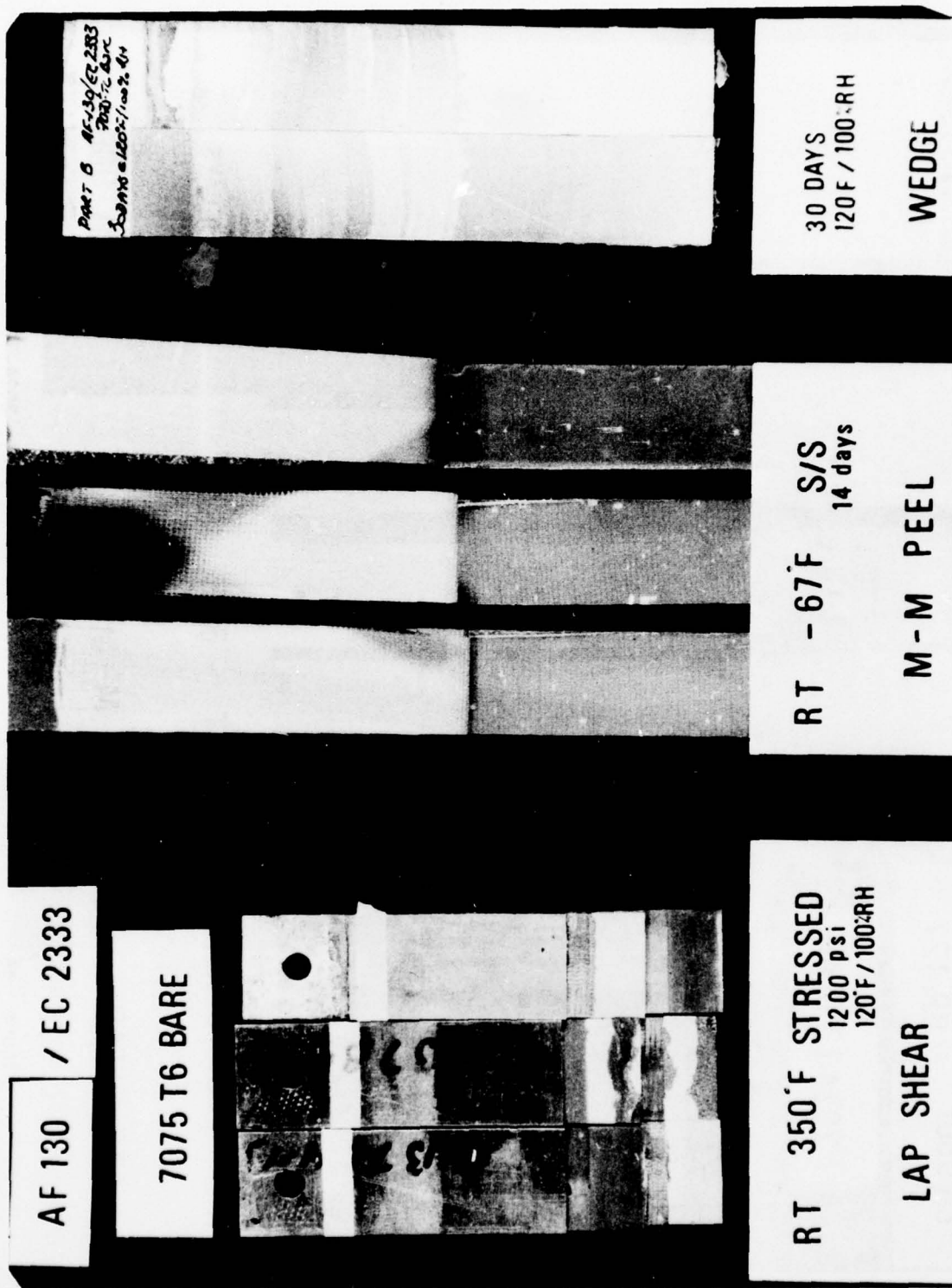


Figure 47 Specimen Bond Surfaces -- AF 130, 7075-T6 Bare

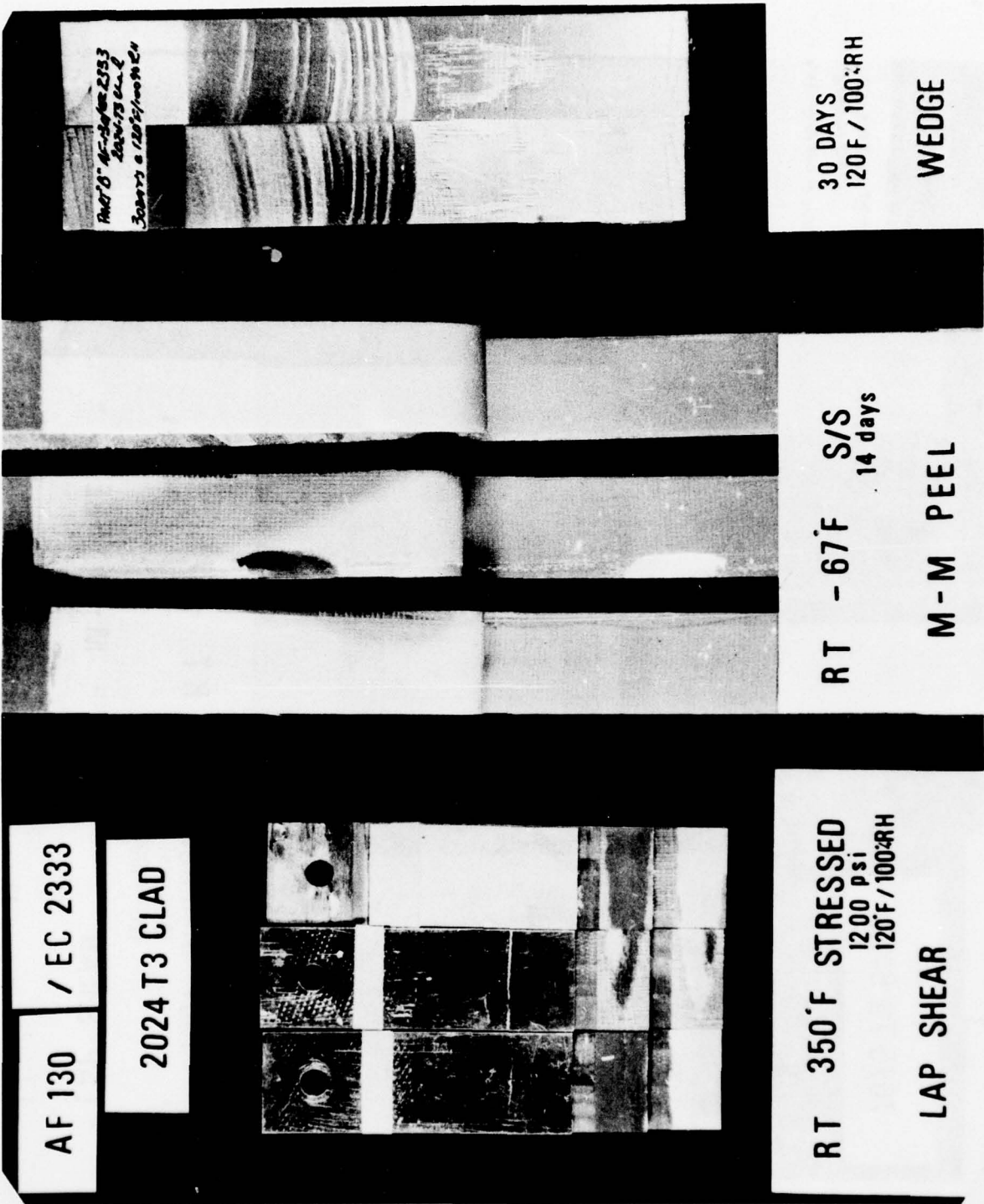
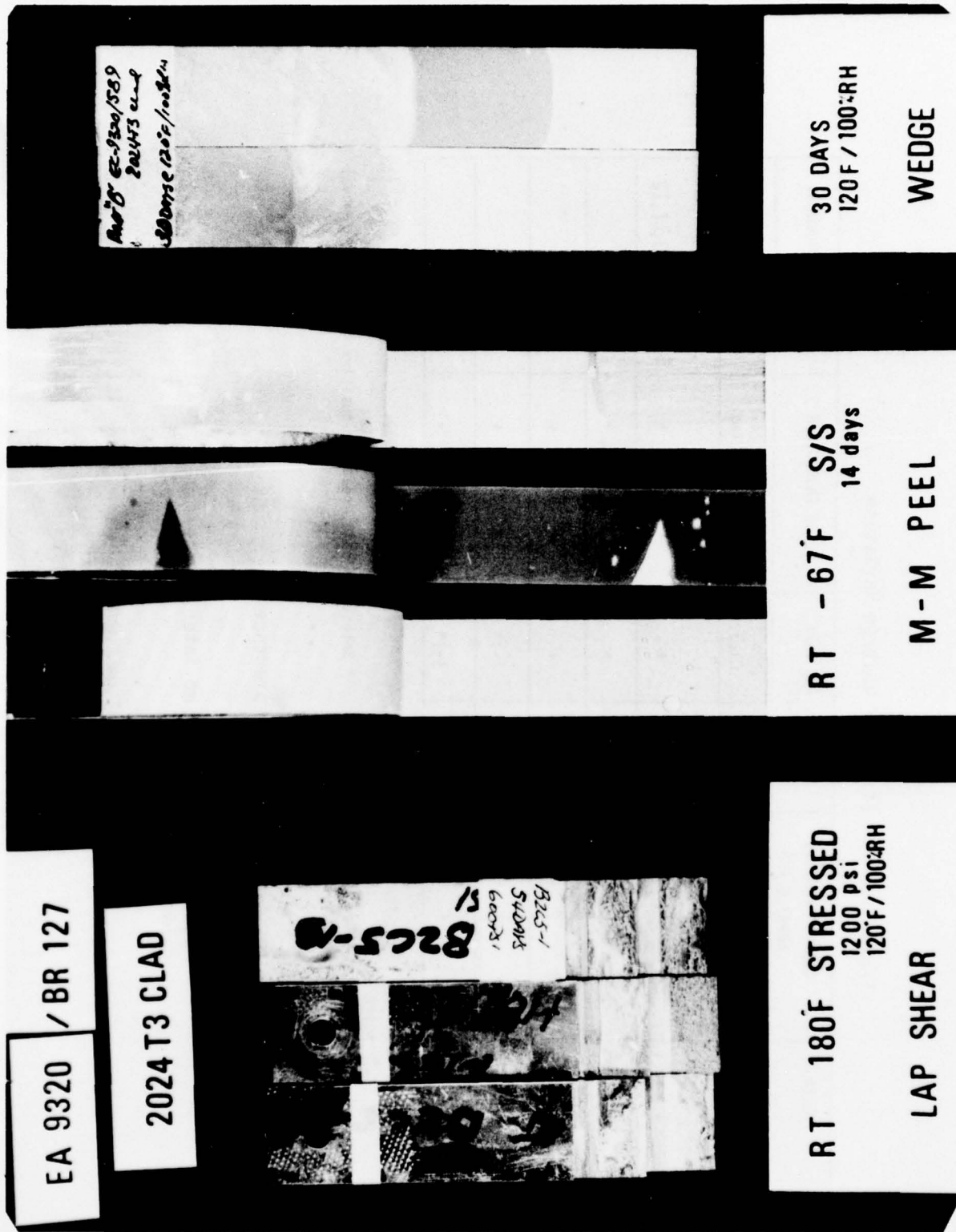


Figure 48 Specimen Bond Surfaces -- AF 130, 2024-T3 Clad



Figure 49 Specimen Bond Surfaces -- EA 9320, 7075-T6 Bare



Specimen Bond Surfaces -- EA 9320, 2024-T3 Clad

Figure 50

Table 1 Effect of Voltage Variation

VOLTAGE	CURRENT DENSITY AMPS/.5 FT ²		3M Co. 250 TAPE TEST		COLOR INSPECTION		OXIDE THICKNESS	
	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad
DC VOLTS								
1	.74	.58	-	+	+	+	See Figs. 10, 11, 12	
2	1.2	.78	+	+	+	+		
4	1.70	1.40	+	+	+	+		
6	2.0	1.80	(-)*	(-)*	+	+		
10	4.6	4.2	(-)*	(-)*	+	+		

NOTES:

RT (70-75°F) condition
PR 50 Paste (10-12% conc.)
10 minutes

* Retests in same areas are positive due to smut removal

+ = positive tape test or presence of
interference color

- = negative tape test or no interference
color

Table 2 Voltage Variation Wedge Test Summary

VOLTAGE D.C. VOLTS	7075-T6 BARE 120°F/100% RH										2024-T3 CLAD 120°F/100% RH						
	Initial Length Inches	CRACK GROWTH, INCHES					Initial Length, Inches	CRACK GROWTH, INCHES					1 HR	4 HR	24 HR	14 Day	30 Day
		1 HR	4 HR	24 HR	14 Day	30 Day		1 HR	4 HR	24 HR	14 Day	30 Day					
1	1.53	2.26	Failed	Failed			1.49	0.00	0.00	0.07	0.07	0.07	0.00	0.00	0.07	0.07	0.07
	1.58	0.36	3.10	Failed			1.39	"	"	0.09	0.09	0.09	"	"	0.09	0.09	0.09
	1.45	0.79	2.75	"			1.42	"	"	0.00	0.00	0.08	"	"	0.00	0.08	0.08
	1.47	0.53	3.00	"			1.36	"	"	0.10	0.10	0.10	"	"	0.10	0.10	0.10
	1.53	0.20	3.30	"			1.41	"	"	0.08	0.08	0.08	"	"	0.08	0.08	0.08
2	1.57	0.00	0.00	0.05	0.10	0.10	1.45	0.04	0.04	0.04	0.09	0.09	0.04	0.04	0.04	0.09	0.09
	1.47	"	"	0.05	0.05	0.05	1.30	0.00	0.00	0.06	0.06	0.06	"	"	0.06	0.06	0.06
	1.54	"	"	0.05	0.05	0.17	1.37	"	"	0.04	0.04	0.04	"	"	0.04	0.04	0.04
	1.63	"	"	0.00	0.05	0.05	1.32	"	"	0.04	0.04	0.04	"	"	0.04	0.04	0.04
	1.64	"	"	0.04	0.04	0.04	1.39	"	"	0.04	0.04	0.04	"	"	0.04	0.04	0.04
4	1.63	0.00	0.00	0.06	0.06	0.06	1.36	0.00	0.00	0.09	0.09	0.09	0.00	0.00	0.09	0.09	0.09
	1.47	"	"	0.06	0.06	0.06	1.36	"	"	0.06	0.06	0.06	"	"	0.06	0.06	0.06
	1.48	"	"	0.06	0.06	0.06	1.32	"	"	0.07	0.07	0.07	"	"	0.07	0.07	0.07
	1.52	"	"	0.09	0.09	0.09	1.33	"	"	0.08	0.08	0.08	"	"	0.08	0.08	0.08
	1.53	"	"	0.06	0.06	0.06	1.36	"	"	0.04	0.04	0.04	0.04	0.04	0.04	0.12	0.12
6	1.53	0.00	0.00	0.07	0.07	0.14	1.44	0.03	0.03	0.03	0.10	0.10	0.03	0.03	0.03	0.10	0.10
	1.53	"	"	0.07	0.07	0.07	1.39	0.04	0.04	0.04	0.09	0.09	0.04	0.04	0.04	0.09	0.09
	1.54	"	"	0.00	0.06	0.06	1.35	0.04	0.04	"	0.10	0.10	"	"	"	0.10	0.10
	1.44	"	"	0.00	0.07	0.18	1.30	0.04	0.04	"	0.10	0.10	0.04	0.04	"	0.10	0.10
	1.57	"	"	0.04	0.04	0.15	1.37	0.04	0.04	"	0.04	0.13	0.04	0.04	0.04	0.13	0.13
10	1.51	0.05	0.00	0.06	0.06	0.11	1.45	0.05	0.05	0.05	0.11	0.11	0.05	0.05	0.05	0.11	0.11
	1.48	0.03	0.03	0.03	0.03	0.12	1.30	0.00	0.00	0.06	0.06	0.06	0.00	0.00	0.06	0.06	0.06
	1.42	0.04	0.04	0.04	0.04	0.12	1.40	"	"	0.00	0.06	0.06	"	"	0.00	0.06	0.06
	1.40	0.05	0.05	0.06	0.05	0.05	1.33	"	"	0.06	0.06	0.06	"	"	0.06	0.06	0.06
	2.01	0.00	0.00	0.00	0.00	0.09	1.34	"	"	0.06	0.06	0.06	"	"	0.06	0.06	0.13

NOTE: FM73/BR127 ADHESIVE/PRIMER

Table 3 Effect of Anodize Temperature

TEMP. OF	VOLTS	CURRENT DENSITY AMPS/.5ft ²		3M Co 250 TAPE TEST		COLOR INSPECTION		OXIDE THICKNESS	
		7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad
102	1	3.2	2.0	-	Trace	-	-	See Figs.15, 16	
95-105	2	3.2-2.8	2.4	-	Trace	-	-	"	
103-108	4	5.8-6.6	4.2-4.8	+	+	+	+	"	
42-48	2	.7	.5	Trace	Trace	Questionable (-)	Questionable (-)	See Figs.17, 18	
46-47	4	1.4	1.2	Trace	Trace	+	+	"	
45	10	2.8	2.0	Trace to Positive	Trace to Positive	+	+	"	

Note:

+ = positive tape test or presence of
interference color

- = negative tape test or no interference
color

Table 4 Anodize Temperature Wedge Test Summary

TEMP	VOLTS	7075-T6 BARE 120°F/100% RH						2024-T3 CLAD 120°F/100% RH					
		Initial Length, Inches	CRACK GROWTH, INCHES					Initial Length, Inches	CRACK GROWTH, INCHES				
			1 HR	4 HR	24 HR	14 DAY	30 DAY		1 HR	4 HR	24 HR	14 DAY	30 DAY
98-110	1	1.67	1.20	2.03	Failed			1.49	0.82	1.69	Failed		
		1.56	1.33	2.18	"			1.38	0.73	1.79	"		
		1.74	0.53	3.21	"			1.47	0.59	1.41	"		
		1.68	1.22	1.76	"			1.43	1.31	3.51	"		
		1.66	1.50	3.23	"			1.57	0.75	1.37	"		
95-105	2	1.62	1.62	Failed				1.44	1.18	2.17	Failed		
		1.62	0.82	1.87	Failed			1.46	3.48	Failed			
		1.51	1.34	1.92	"			1.34	2.23	Failed			
		1.57	0.91	1.88	"			1.44	0.10	0.67	Failed		
		1.58	0.38	3.33	"			1.38	0.72	2.17	Failed		
103-108	4	1.61	0.18	0.18	0.60	0.60	0.67	1.39	0.05	0.05	0.05	0.12	0.12
		1.51	0.00	0.00	0.07	0.33	0.33	1.45	0.00	0.00	0.06	0.06	0.06
		1.48	0.03	0.03	0.10	0.58	0.58	1.35	0.00	0.00	0.00	0.07	0.14
		1.53	0.07	0.07	0.71	0.78	0.78	1.133	0.00	0.00	0.07	0.07	0.16
		1.57	0.25	0.64	1.69	1.69	1.69	1.38	0.00	0.00	0.07	0.07	0.07
48	2	1.53	0.06	0.06	0.06	0.14	0.19	1.43	0.20	1.07	1.13	1.13	1.13
		1.55	0.04	0.04	0.04	0.11	0.17	1.35	0.12	0.27	1.39	1.39	1.39
		1.58	0.04	0.04	0.04	0.11	0.11	1.32	0.14	0.96	1.01	1.01	1.01
		1.55	0.05	0.05	0.05	0.12	0.12	1.33	0.48	1.74	1.74	1.74	1.74
		1.47	0.06	0.06	0.06	0.12	0.24	1.41	0.07	0.07	0.12	0.12	0.24
46	4	1.56	0.04	0.04	0.04	0.10	0.10	1.50	0.05	1.15	1.15	1.15	1.15
		1.46	0.04	0.04	0.04	0.16	0.16	1.34	0.23	0.70	1.08	1.08	1.08
		1.44	0.04	0.04	0.04	0.18	0.18	1.37	0.20	0.59	1.54	1.54	1.54
		1.46	0.07	0.07	0.07	0.13	0.13	1.32	0.25	0.66	1.68	1.68	1.68
		1.53	0.07	0.07	0.07	0.14	0.14	1.38	0.11	0.23	1.36	1.36	1.36
45	10	1.53	0.06	0.06	0.06	0.13	0.13	1.43	0.07	0.07	0.19	0.19	0.27
		1.52	0.001	0.00	0.04	0.09	0.09	1.37	0.18	0.77	0.77	0.77	0.77
		1.53	0.00	0.00	0.05	0.05	0.12	1.42	0.14	1.31	1.46	1.46	1.46
		1.54	0.00	0.00	0.00	0.06	0.06	1.41	0.33	1.56	1.56	1.56	1.56
		1.50	0.00	0.00	0.05	0.05	0.05	1.39	0.22	0.69	1.91	1.91	1.91

Table 5 Effect of Anodizing Time

TIME MIN	CURRENT DENSITY AMPS/.5ft ²		3M Co. 250 TAPE TEST		COLOR INSPECTION		OXIDE THICKNESS	
	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad
1	1.2	1.2	-	-	-		See Figs.20, 21	
5	1.4	1.2	+	Trace to Negative	+	+	"	"
15	1.4	1.2	+	Trace to Negative	+	+	"	"
20	1.4	1.2	+	+	+	+	"	"

NOTES:

Constant voltage - 4 volts
PR 50 Paste (10-12% phosphoric acid)
Temperature - RT (70-75°F)

+ = positive tape test or presence of
interference color

- = negative tape test or no interference
color

Table 6 Anodizing Time Wedge Test Summary

ANODIZING TIME MINUTE	7075-T6 BARE 120°F/100% RH						2024-T3 CLAD 120°F/100% RH					
	Initial Length, Inches	CRACK GROWTH, INCHES					Initial Length, Inches	CRACK GROWTH, INCHES				
		1 HR	4 HR	24 HR	14 DAY	30 DAY		1 HR	4 HR	24 HR	14 DAY	30 DAY
1	1.58 1.56 1.57 1.62 1.42	2.24 1.84 0.00 0.92 0.53	Failed Failed 0.22 1.93 0.53	Failed Failed Failed Failed			1.46 1.43 1.47 1.48 1.43	2.86 2.88 2.77 2.86 2.80	Failed " " " "			
5	1.73 1.53 1.58 1.46 1.61	0.00 " " " "	0.00 " " " "	0.07 0.00 0.00 0.07 0.06		0.07 0.00 0.00 0.07 0.06	1.41 1.44 1.37 1.37 1.41	0.07 0.11 0.00 0.00 0.65	1.29 1.29 0.00 0.00 1.10	1.29 1.29 0.00 0.00 1.10	1.41 1.37 0.00 0.07 1.10	1.53 1.43 0.08 0.07 1.10
15	1.56 1.73 1.57 1.58 1.57	0.00 " " " "	0.00 0.05 0.05 0.05 0.05	0.10 0.05 0.12 0.11 0.13		0.10 0.05 0.12 0.11 0.18	1.44 1.45 1.42 1.41 1.48	0.00 " " " "	0.00 " " " "	0.05 0.05 0.03 0.06 0.00	0.05 0.05 0.03 0.06 0.08	0.05 0.05 0.03 0.06 0.08
20	1.63 1.52 1.58 1.48 1.50	0.00 " " " "	0.00 " " " "	0.05 0.00 0.00 0.00 0.06		0.10 0.04 0.00 0.11 0.06	1.45 1.51 1.38 1.47 1.46	0.00 " " " "	0.00 " " " "	0.00 0.06 0.00 0.00 0.00	0.07 0.12 0.08 0.00 0.07	0.07 0.12 0.08 0.10 0.07

NOTES:

- : 70-750F
- : PR50 paste (10-12% phosphoric acid)
- : 4 volts
- : FM 73/BR127

Table 7 Effect of Rinse Delay

RINSE DELAY MINUTES	CURRENT DENSITY AMPS/.5 ft ²		3M Co. 250 TAPE TEST		COLOR INSPECTION		OXIDE THICKNESS	
	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad	7075-T6 Bare	2024-T3 Clad
2	1.5	1.2	+	Trace to Negative	Faint	+	See Figs. 23, 24	
5	1.6	1.3	+	Trace to Negative	Decreasing (very faint)	+	"	
10	1.6	1.3	Trace to Negative	Negative	-	+	"	
0 From Task A-1	1.7	1.4	+	+	+	+	See Figs. 10, 11, 12	

NOTE: Anodized at 4 volts, 10 minutes, 70-75°F, PR 50 Paste (10-12% phosphoric acid)

+ = positive tape test or presence of
interference color

- = negative tape test or no interference
color

Table 8 Rinse Delay Time Wedge Test Summary

RINSE DELAY MINUTE	7075-T6 BARE 120°F/100% RH						2024-T3 CLAD 120°F/100% RH					
	Initial Length, Inches	CRACK GROWTH, INCHES					Initial Length, Inches	CRACK GROWTH, INCHES				
		1 HR	4 HR	24 HR	14 DAY	30 DAY		1 HR	4 HR	24 HR	14 DAY	30 DAY
2	1.56 1.57 1.71 1.47 1.48	0.00 " " " "	0.00 " " " "	0.08 0.00 0.00 0.00 0.05	0.16 0.06 0.00 0.07 0.05	0.16 0.06 0.00 0.07 0.12	1.51 1.44 1.52 1.34 1.38	0.00 " " " "	0.00 " " " "	0.07 0.00 0.00 0.00 0.08	0.07 0.07 0.00 0.00 0.08	0.07 0.07 0.00 0.00 0.08
5	1.62 1.65 1.75 1.54 1.53	0.00 " " " "	0.00 " " " "	0.00 0.00 0.00 0.00 0.00	0.05 0.07 0.00 0.08 0.08	0.11 0.07 0.09 0.08 0.08	1.38 1.33 1.37 1.36 1.43	0.00 " " " "	0.00 " " " "	0.05 0.05 0.05 0.05 0.05	0.05 0.13 0.05 0.12 0.05	0.12 0.13 0.11 0.12 0.09
10	1.50 1.54 1.58 1.74 1.44	0.00 0.00 0.00 1.87 0.60	0.00 0.00 2.15 2.28 0.60	0.06 0.73 Failed Failed 0.60	0.06 0.73 0.73 1.05 1.05	0.06 0.73 0.73 1.05 1.05	1.38 1.43 1.42 1.42 1.37	0.00 " " " "	0.00 " " " "	0.07 0.00 0.00 0.00 0.00	0.14 0.06 0.09 0.00 0.09	0.14 0.06 0.09 0.08 0.09
No Delay	SEE TABLE 2, DATA FOR 4 VOLTS											

NOTE: Anodize at 4 volts, 10 minutes, 70-75°F, PR 50 Paste (10-12% phosphoric acid)
FM73/BR127

Table 9 Effect of Part Size

ELECTRICAL CONTACTS	CURRENT DENSITY, AMPS/FT²	TAPE TEST	COLOR INSPECTION
4	1.45	+	+
3	1.4	+	+
2	1.3	+	+
1	1.2	+	+

Note:

+ = positive tape test or presence of
interference color

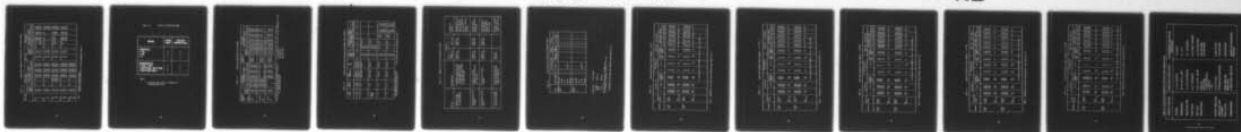
AD-A056 606

BOEING COMMERCIAL AIRPLANE CO SEATTLE WASH
ANODIZE OPTIMIZATION AND ADHESIVE EVALUATION FOR REPAIR APPLICA--ETC(U)
JAN 78 M C LOCKE, R E HORTON, J E MCCARTY F33615-73-C-5171
AFML-TR-78-7 NL

UNCLASSIFIED

2 OF 2

AD
A056606



END
DATE
FILMED
8-78
DDC

Table 10 Wedge Test Summary of Large (24- x 24-inch) Panel

SPEC. NO.	120°F/100%RH							SPEC. NO.	120°F/100%RH						
	Initial Length Inches	CRACK GROWTH - INCHES					Initial Length Inches		CRACK GROWTH - INCHES						
		1 HR	4 HR	24 HR	14 DAY	30 DAY			1 HR	4 HR	24 HR	14 DAY	30 DAY		
A3-1	1.58	0.00	0.00	0.00	0.06	0.06	A3-26	1.47	0.00	0.00	0.06	0.06	0.06	0.06	0.06
2	1.52	"	"	0.07	0.07	0.07	27	1.46	"	"	0.06	0.06	0.06	0.06	0.06
3	1.50	"	"	0.07	0.07	0.07	28	1.61	"	"	0.05	0.05	0.05	0.05	0.05
4	1.49	"	"	0.06	0.06	0.06	29	1.51	"	0.03	0.03	0.03	0.08	0.08	0.08
5	1.47	"	"	0.06	0.06	0.06	30	1.47	"	0.05	0.05	0.05	0.11	0.11	0.11
A3-6	1.47	0.00	0.00	0.06	0.14	0.14	A3-31	1.61	0.00	0.00	0.00	0.00	0.00	0.00	0.00
7	1.48	"	"	0.00	0.00	0.08	32	1.48	"	"	"	"	"	"	0.00
8	1.46	"	"	0.06	0.06	0.12	33	1.57	"	"	"	"	"	"	0.03
9	1.49	"	"	0.06	0.06	0.12	34	1.58	"	"	"	"	"	"	0.00
10	1.46	"	"	0.06	0.06	0.12	35	1.48	"	"	"	"	"	"	0.00
A3-11	1.54	0.00	0.00	0.08	0.08	0.08	A3-36	1.29	0.00	0.00	0.00	0.00	0.00	0.00	0.00
12	1.57	"	0.00	0.00	0.09	0.09	37	1.43	"	0.05	0.05	0.05	0.05	0.05	0.05
13	1.46	"	0.06	0.06	0.06	0.06	38	1.47	"	0.00	0.00	0.00	0.00	0.00	0.00
14	1.39	"	0.05	0.05	0.05	0.05	39	1.46	"	"	"	"	"	"	0.00
15	1.49	"	0.06	0.06	0.06	0.06	40	1.51	"	"	"	"	"	"	0.00
A3-16	1.49	0.00	0.00	0.07	0.07	0.07	A3-41	1.49	0.00	0.06	0.06	0.06	0.06	0.06	0.13
17	1.57	"	"	0.00	0.08	0.08	42	1.57	"	0.00	0.00	0.00	0.00	0.00	0.00
18	1.58	"	"	0.00	0.08	0.08	43	1.52	"	"	0.06	0.06	0.12	0.19	0.19
19	1.46	"	"	0.06	0.06	0.06	44	1.54	"	"	0.06	0.06	0.12	0.16	0.16
20	1.48	"	"	0.07	0.06	0.06	45	1.63	"	"	0.00	0.00	0.00	0.00	0.00
A3-21	1.47	0.00	0.00	0.04	0.04	0.09									
22	1.53	"	"	0.04	0.04	0.04									
23	1.42	"	"	0.04	0.04	0.04									
24	1.58	"	"	0.05	0.05	0.05									
25	1.51	"	"	0.07	0.07	0.12									

NOTE: : FM73/BR127 Adhesive/Primer
: 4 Volts, 10 Minutes, PR50 Paste (10-12% phosphoric acid)

Table 11

Effect of Anodizing Mode

MODE	TAPE TEST	COLOR INSPECTION
VERTICAL <ul style="list-style-type: none">• TB• 3S• T	<ul style="list-style-type: none">+++	<ul style="list-style-type: none">+++
HORIZONTAL <ul style="list-style-type: none">• TOP - TOP• BOTTOM - BOTTOM• BOTTOM ONLY	<ul style="list-style-type: none">+++	<ul style="list-style-type: none">+++

Note:

+ = positive tape test or presence of interference color

Table 12 Anodizing Mode Wedge Test Summary

ANODIZING CONFIG.	Initial Length Inches	120°F/100%RH CRACK GROWTH - INCHES					ANODIZING CONFIG	Initial Length Inches	120°F/100%RH CRACK GROWTH - INCHES				
		CRACK GROWTH - INCHES							CRACK GROWTH - INCHES				
		1 HR	4 HR	24 HR	14 DAY	30 DAY			1 HR	4 HR	24 HR	14 DAY	30 DAY
① Vertical (TB)	1.64	0.00	0.00	0.07	0.07	0.07	Horizontal (upper surfaces)	1.47	0.00	0.00	0.05	0.05	0.05
	1.55	0.23	0.23	0.23	0.23	0.23		1.43	0.00	0.00	0.06	0.06	0.06
	1.48	0.51	0.51	0.51	0.51	0.51		1.57	0.00	0.00	0.00	0.03	0.03
	1.43	0.00	0.00	0.09	0.09	0.09		1.61	0.00	0.00	0.00	0.06	0.06
	1.47	0.00	0.00	0.10	0.17	0.17		1.48	0.00	0.00	0.07	0.07	0.07
Vertical (3S)	1.47	0.00	0.07	0.14	0.14	0.14	Horizontal (Lower surfaces)	1.54	0.06	0.06	0.06	0.14	0.14
	1.41	0.20	0.20	0.28	0.28	0.38		1.47	0.00	0.00	0.05	0.11	0.11
	1.39	2.78	Failed					1.42	0.00	0.00	0.00	0.10	0.10
	1.47	3.73	Failed					1.51	0.00	0.00	0.07	0.15	0.15
	1.43	0.08	0.40	0.44	0.44	0.44		1.69	1.33	Failed			
Vertical (T)	1.71	0.00	0.00	0.07	0.07	0.07	Horizontal (Lower surfaces)	1.58	2.92	Failed			
	1.67	"	"	0.00	0.07	0.07		1.57	0.21	0.00	0.00	0.00	0.07
	1.67	"	"	0.00	0.08	0.08		1.56	0.00	0.00	0.00	0.09	0.09
	1.49	"	"	0.06	0.06	0.13		1.50	0.00	0.00	0.00	0.10	0.10
	1.62	"	"	0.00	0.11	0.11		1.69	0.00	0.00	0.00	0.11	0.11

NOTE: ① TB = Secured Top and Bottom of Screen/Gauze
 3S = Secured Top and 2 Sides
 T = Secured Top Edge Only
 4 volts, 10 minutes, PR 50 paste (10-12% phosphoric acid)
 R.T. (70-75°F)
 FM 73/BR 127
 7075-T6 Bare

Table 13 Battery Anodizing Screening and Wedge Test Summary

D.C. VOLTS	AMPS/ 0.5 Ft ²	COLOR INSPECTION	TAPE TEST	INITIAL CRACK LENGTH INCHES	120°F/100% RH EXPOSURE					
					CRACK GROWTH INCHES					
					1 HR	4 HR	24 HR	14 DAY	30 DAY	
2*	0.05	Negative	Negative							
2*	0.04	Negative	Negative							
4	0.5	Questionable	Negative	1.46 1.54 1.53 1.62 1.68	2.84 2.59 2.57 2.67 3.56	Failed " " " "				
4 (Second Assy)	0.5	Questionable	Negative	1.49 1.53 1.47 1.47 1.43	2.49 3.69 3.01 3.00 3.78	Failed " " " "				
6	1.8	Positive	Positive	1.49 1.50 1.54 1.56 1.53	0.00 " " " "	0.00 " " " "	0.04 0.00 " " "	0.10 0.11 0.06 0.06 0.09	0.10 0.11 0.06 0.06 0.09	
12	2.6	Positive	Positive	1.47 1.38 1.37 1.48 1.52	0.00 " " " "	0.00 " " " "	0.06 0.00 " " "	0.13 0.11 0.07 0.09 0.09	0.13 0.11 0.07 0.09 0.09	



* Smut appearance on anodized surface,
not bonded into wedge assemblies

PR 50 paste (10-12% phosphoric acid)

Table 14. Common Errors and Corrective Action

Errors	Observations	Results		Corrective Action
		Color Inspection	Tape Test	
o Reverse leads, (+) on screen, (-) on aluminum	o Vigorous reaction seen on the screen surface; current density surge to extremely high level at same voltage	Negative color test	Negative tape test	Immediately turn off rectifier; check lead for (+) on aluminum and (-) on screen; restart anodize
o Screen contacting aluminum	o Surge in current density; spot burning on aluminum	Negative color test	Negative tape test	Extend gauze beyond periphery of screen
o Inadequate gelled acid	o Dry spots on gauze/screen	Negative color test	Negative tape test	Saturate gauze with gelled acid; reapply gelled acid over screen
o Too high voltage (over 10 volts)	o High current density (over 10 amps/sq ft); vigorous reaction	Smut on surface		Maintain voltage in the 6-volt range (4-6 volts)

Table 15 Test Matrix for Bond Verification Tests

Alloy	Adhesive 	0.5-inch Lap Shear			Wedge	M/M Peel		
		RT	180°F	1200 psi Stress 		120°F/100% RH	RT	-67°F
2024-T3 Clad	AF 127-3	x	x	x	x	x	x	x
	EA 9601	x	x	x	x	x	x	x
	FM 73	x	x	x	x	x	x	x
	AF 130	x	(350°F)	x	x	x	x	x
	EA 9320	x	x	x	x	x	x	x
7075-T6 Bare	AF 127-3	x	x	x	x	x	x	x
	EA 9601	x	x	x	x	x	x	x
	FM 73	x	x	x	x	x	x	x
	AF 130	x	(350°F)	x	x	x	x	x
	EA 9320	x	x	x	x	x	x	x

¹ EA 9320 - RT cure system

AF 127-3

EA 9601 - 250°F cure

FM 73

AF 130 - 350°F cure

² 120°F/100% RH exposure, 4 specimens each
all others are 5 specimens. 600 psi for EA 9320

Phosphoric Acid Non-Tank Anodize (see Section 3.1)

Table 16 Bond Verification Tests Summary -- FM 73

ADHEREND	ADHESIVE/ PRIMER	LAP SHEAR, PSI			METAL-METAL PEEL lb. in./in.			WEDGE TEST -120°F/100% RH				
		RT	180°F	1200 psi* SUSTAINED STRESS	RT	-67°F	14 DAY SALT SPRAY	INITIAL LENGTH INCHES	CRACK GROWTH, INCHES			
									1 HR	4 HR	24 HR	14 DAY
204T3 CLAD	FM73/ BR127	4640	3740	4380	63	55	68	1.37	0.06	0.06	0.11	0.16
		4760	3660	4520	67	60	68	1.26	0.0	0.06	0.07	0.13
		4680	3540	4440	69	61	54	1.43	0.0	0.0	0.07	0.11
		4680	3400	4260	66	58	65	1.38	0.0	0.06	0.06	0.13
		4580	3480		67	61	65	1.46	0.0	0.05	0.05	0.12
7075T6 BARE	AVE. STD. DEV.	4670 65	3560 139	4400 109	66 2	59 2.5	64 5.8	1.38	0.012	0.046	0.072	0.096
												0.13
	FM73/ BR127	4940	3792	4740	51	40	53	1.47	0.05	0.05	0.05	0.16
		5040	3948	4830	51	43	53	1.44	0.0	0.0	0.06	0.13
		5220	3820	5100	51	43	50	1.48	0.0	0.0	0.0	0.09
		5000	3626	4880	51	48	53	1.38	0.0	0.0	0.05	0.12
		5200	3908		51	42	50	1.64	0.0	0.0	0.05	0.14
	AVE. STD. DEV.	5080 124	3818 125	4900 148	51 0	41.6 1.5	51.8 1.6	1.48	0.01	0.01	0.042	0.11
												0.14
				* 60 days exposure								
				120°F/100%RH no failure - test for residual								

Note: Phosphoric Acid Non-Tank Anodize (see Section 3.1)

Table 17 Bond Verification Tests Summary -- AF 127-3

ADHEREND	ADHESIVE/ PRIMER	LAP SHEAR PSI			METAL-METAL PEEL lb. in./in.			WEDGE TEST -120°F/100% RH					
		RT	180°F	1200 psi* SUSTAINED STRESS	RT	-67°F	14 DAY SALT SPRAY	INITIAL LENGTH INCHES	CRACK GROWTH, INCHES				
									1 HR	4 HR	24 HR	14 DAY	30 DAY
2024T3 CLAD	AF127-3/ BR127	4440	2780	3724	78	16	72	1.32	0.22	0.22	0.22	0.32	0.48
		4520	2740	3784	78	16	75	1.37	0.16	0.16	0.16	0.28	0.38
		4400	2664	3372	78	18	75	1.34	0.15	0.20	0.20	0.27	0.39
		4480	2676	3676	80	21	75	1.28	0.13	0.13	0.13	0.26	0.40
		4420	2668		80	37	76	1.33	0.13	0.20	0.20	0.29	0.36
7075T6 BARE	AVE. STD. DEV.	4452 48	2705 51	3639 183	79 1.1	21.6 8.8	74 1.6	1.33	0.16	0.18	0.18	0.28	0.40
	AF127-3/ BR127	5020	2904	3992	60	10	57	1.50	0.15	0.21	0.21	0.28	0.37
		4920	2830	3948	62	10	58	1.43	0.19	0.19	0.19	0.34	0.46
		4820	2870*	4080	62	10	58	1.46	0.18	0.18	0.18	0.29	0.39
		4980	2892	3944	62	10	58	1.43	0.18	0.18	0.18	0.32	0.43
		4920	2864		55	12	58	1.50	0.18	0.23	0.23	0.36	0.44
	AVE. STD. DEV.	4932 75	2870 32	3991 63	60 3	10 1	58 0.5	1.46	0.18	0.20	0.20	0.32	0.42
				* 60 days exposure	120°F/100%RH no failure - test for residual								

Note: Phosphoric Acid Non-Tank Anodize (see Section 3.1)

Table 18 Bond Verification Tests Summary -- EA 9601

ADHEREND	ADHESIVE/ PRIMER	LAP SHEAR PSI			METAL-METAL PEEL lb. in./in.			WEDGE TEST -120°F/100% RH					
		RT	180°F	1200 psi * SUSTAINED STRESS	RT	-67°F	14 DAY SALT SPRAY	INITIAL LENGTH SPRAY	CRACK GROWTH, INCHES				
									1 HR	4 HR	24 HR	14 DAY	30 DAY
2024T3 CLAD	EA9601/ BR127	4700	3640	4800	57	10	60	1.64	0.08	0.08	0.13	0.19	0.27
		4820	3584	4800	57	9	40	1.63	0.09	0.09	0.16	0.23	0.31
		4680	3492	4860	57	6	39	1.57	0.08	0.08	0.16	0.23	0.23
		4800	3496	4780	54	10	38	1.58	0.08	0.03	0.14	0.22	0.22
		4720	3428		55	7	44	1.55	0.08	0.15	0.15	0.24	0.24
7075T6 BARE	AVE. STD. DEV.	4744	3528	4810	56	8.4	44.2	1.59	0.08	0.10	0.15	0.22	0.25
		62	83	34	1.4	1.9	9.1						
		5260	3636	5300	37	7	45	1.69	0.11	0.11	0.17	0.26	0.26
		5320	3664	5280	42	4	45	1.65	0.12	0.12	0.12	0.25	0.34
		5200	3676	5280	40	4	45	1.76	0.11	0.11	0.16	0.28	0.28
	AVE. STD. DEV.	5440	3600	5240	40	4	45	1.64	0.09	0.09	0.18	0.18	0.33
		5480	3744		39	9	45	1.73	0.10	0.14	0.14	0.24	0.30
		5340	3664	5275	39	5.6	45	1.69	0.11	0.11	0.15	0.24	0.30
		118	53	25	2	2.3	0						
					* 60 days exposure			120°F/100%RH no failure - test for residual					

Note: Phosphoric Acid Non-Tank Anodize (see Section 3.1)

Table 19 Bond Verification Tests Summary -- AF-130

ADHEREND	ADHESIVE/ PRIMER	LAP SHEAR PSI			METAL-METAL PEEL lb. in./in.			WEDGE TEST - 120°F/100% RH					
		RT	350°F	1200 psi* SUSTAINED STRESS	RT	-67°F	14 DAY SALT SPRAY	INITIAL LENGTH INCHES	CRACK GROWTH, INCHES				
									1 HR	4 HR	24 HR	14 DAY	30 DAY
2024T3 CLAD	AF130/ EC2333	1540	1884	3180	3.6	12.0	8.0	2.57	0.0	0.0	0.0	0.0	0.0
		1630	2064	3080	3.6	13.5	8.0	2.56	0.0	0.0	0.0	0.0	0.0
		1672	2096	3104	3.6	12.0	8.0	2.48	0.0	0.0	0.0	0.0	0.0
		1880	2340	2796	3.6	10.5	8.0	2.54	0.0	0.0	0.0	0.0	0.0
		1708	1988		3.6	12.0	8.0	2.43	0.0	0.0	0.0	0.0	0.0
7075T6 BARE	AVE. STD. DEV.	1668	2074	3040	3.6	12.0	8.0	2.52	0.0	0.0	0.0	0.0	0.0
		125	169	168	0	1.0	0						
		1532	1832	3192	3.3	9.0	8.0	2.67	0.0	0.0	0.0	0.0	0.0
		1700	1820	3296	3.0	10.5	8.0	2.89	0.0	0.0	0.0	0.0	0.0
		1656	1748	3212	3.0	12.0	8.0	2.96	0.0	0.0	0.0	0.0	0.0
	AVE. STD. DEV.	1716	1712	3276	3.6	12.0	8.0	2.94	0.0	0.0	0.0	0.0	0.0
		1628	1976		3.9	7.5	8.0	2.78	0.0	0.0	0.0	0.0	0.0
		1646	1817	3244	3.4	10.2	8.0	2.85	0.0	0.0	0.0	0.0	0.0
		72	101	49	0.4	1.9	0						
				* 60 days	exposure	120°F/100%RH	no failure	- test for residual					

Note: Phosphoric Acid Non-Tank Anodize (see Section 3.1)

Table 20 Bond Verification Tests Summary -- EA 9320

ADHEREND	ADHESIVE PRIMER	LAP SHEAR PSI		METAL-METAL PEEL lb. in./in.			WEDGE TEST -120°F/100% RH						
		RT	180°F	600 psi * SUSTAINED STRESS	RT	-67°F	14 DAY SALT SPRAY	INITIAL LENGTH INCHES	CRACK GROWTH, INCHES				
									1 HR	4 HR	24 HR	14 DAY	30 DAY
2024T3 CLAD	EA9320/BR127	3628	460	3992	60	7	65	1.28	0.07	0.07	0.07	0.49	0.68
		3144	828	3000	60	7	50	1.30	0.07	0.07	0.23	0.63	0.75
		3264	832	3644	55	7	55	2.15	0.0	0.0	0.0	0.00	0.00
		3696	716	FAILED	58	6	60	1.34	0.05	0.05	0.13	0.47	0.59
		3748	540	54 DAYS	55	7	58	1.45	0.07	0.07	0.07	0.42	0.58
7075T6 BARE	AVE. STD. DEV.	3496	775	3545	57.6	7	57	1.34	0.06	0.06	0.13	0.50	0.65
		273	157	503	2.5	0.5	5.4						
		3996	476	3544	48	4.5	50	1.52	0.10	0.10	0.10	0.47	0.63
		3592	484	3600	48	4.5	48	1.44	0.13	0.13	0.13	0.42	0.56
		3340	576	3712	50	4.5	50	1.73	0.07	0.07	0.07	0.15	0.25
	AVE. STD. DEV.	3628	580	3664	40	4.5	48	1.39	0.12	0.12	0.12	0.34	0.50
		3488	608		55	4.5	40	1.50	0.08	0.08	0.18	0.38	0.54
		3608	544	3630	48.2	4.5	47	1.52	0.12	0.12	0.12	0.29	0.50
		243	60	73	5.4	0	4						
				* 60 days exposure 120°F/100%RH no failure except as noted - test for residual									

Note: Phosphoric Acid Non-Tank Anodize (see Section 3.1)

Table 21 Summary of Phosphoric Acid Non-Tank Anodize Process

PROCESS VARIABLE	CONDITIONS INVESTIGATED	RECOMMENDED PROCESSING CONDITIONS
VOLTAGE	1, 2, 4, 6, 10 VOLTS	4-6 VOLTS
TEMPERATURE	40, RT (70-75), 100°F	70 - 75° F
ANODIZING TIME	1, 5, 10, 15, 20 MINUTES	10 - 15 MINUTES
RINSE DELAY	0, 2, 5, 10 MINUTES	LESS THAN 5 MINUTES
PART SIZE	6"x6", 6"x12", 24"x24"	ALL SATISFACTORY
ANODIZE MODE	VERTICAL HORIZONTAL o BOTH SURFACES o BOTTOM o TOP	SATISFACTORY SATISFACTORY
BATTERY ANODIZING	2, 4, 6, 12 VOLTS	6 AND 12 VOLTS
ANODIZING OVER FASTENERS	TITANIUM AND ALUMINUM	SATISFACTORY
BARE VS CLAD	7076T6 BARE AND 2024T3 CLAD	SATISFACTORY
COMMON ERROR	4 CONDITIONS IDENTIFIED	CORRECTIVE PROCEDURES PROVIDED